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Official Publication of the Proceedings of the American Association of Textile Chemists and Colorists

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American Dyestuff Reporter was founded in 1917 and the Textile Colorist and Converter (formerly Textile Colorist) was incorporated with it in January, 1949. Published every other Monday by the **HOWES PUBLISHING COMPANY, INC., 44 E. 23rd Street, New York 10, N. Y. C R Howes,** President; **Ernest J Finan,** Executive Vice-President; **Norman A Johnson,** Vice-President & Treasurer; **Myron D Reeser,** Vice-President; **C Essenbreis.** Secretary

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July 7, 1952
VOLUME 41 NUMBER 14

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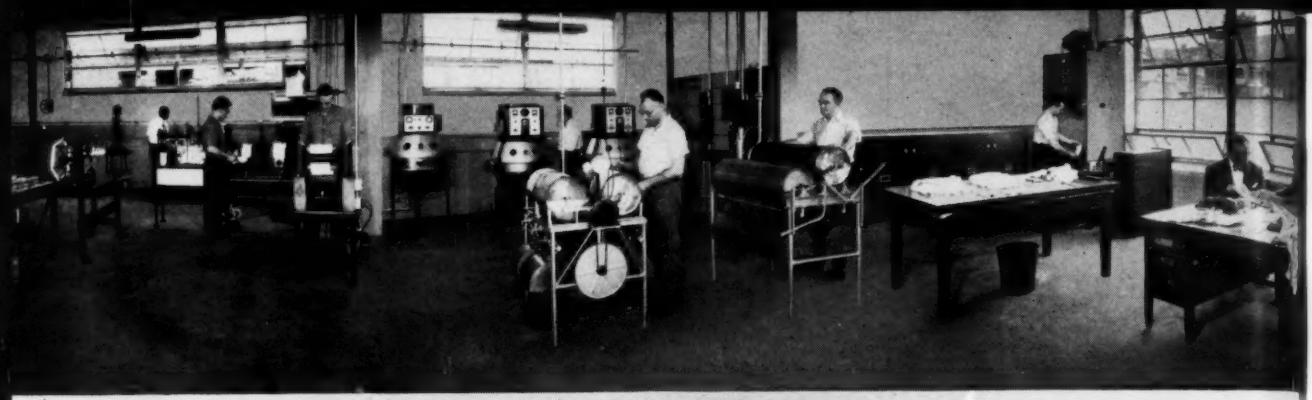

you should make use of our new, modern

service laboratories

to help you with your dyeing problems. There we apply Althouse original ideas to them, together with our latest modern methods of fabric dyeing and our exclusive, built-to-order equipment. We'll come up with a practical, workable solution to enable you to use your own dyehouse techniques. Make use of this free service. A few of our new, model dyeing laboratory departments are illustrated.

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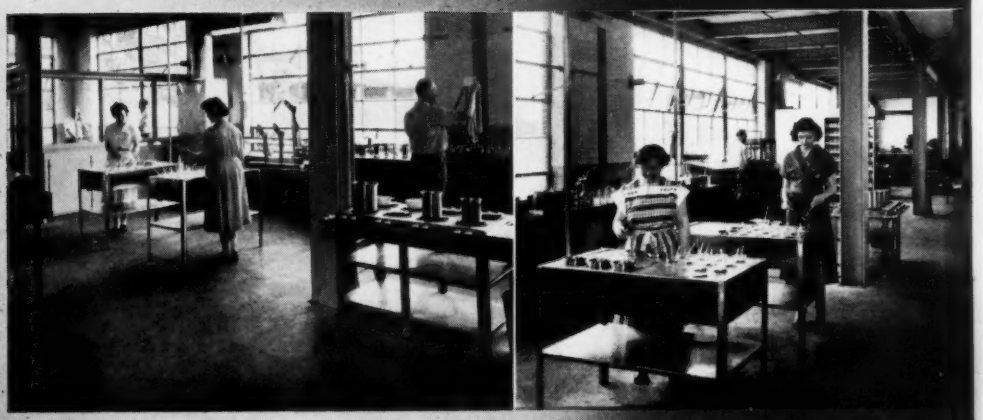
Pace-setter in precision dye-making



AMERICAN
DYESTUFF REPORTER

VOLUME 41 JULY 7, 1952 NUMBER 14

THE EFFECTS OF MAGNESIUM IONS ON THE DETERGENCY OF WOOL USING SODIUM DODECYLTOLUENESULFONATE

T EARL JORDAN, GEORGE VOLZ, JACK GELB and CYRIL ROMANOVSKY
Publicker Industries Inc

INTRODUCTION

THE advantages of alkylarylsulfonates as detergents are common knowledge. In hard water especially, the cleaning properties of these types of compounds for wool are outstanding. Since calcium and magnesium ions are in hard water, the effects of these ions on detergency phenomena are of interest. A search of the literature showed numerous references to the use of calcium and magnesium in cleaning compositions. Only a few appear pertinent. Magnesium salts of sulfonated alcohols have been reported as cleaning compounds *(1)*. Zizinia and McKenna *(6)* included magnesium hydroxide as a base for neutralizing alkylarylsulfonates to form a detergent composition. Miles and Ross *(4)* attributed certain poor detergent properties to the presence of calcium salts in synthetic detergent mixtures. Harris *(2, 3)* found that magnesium sulfate or magnesium chloride is beneficial in a detergent composition.

As a detergent for wool, a mixture of magnesium salts, sodium dodecyltoluenesulfonate, and sodium sulfate was found to be superior to mixtures wherein the magnesium was replaced with calcium, zinc, etc, or wherein the sodium dodecyltoluenesulfonate was replaced by sodium dodecylbenzene, sodium nonylnaphthalenesulfonate, etc. The superiority of the magnesium salts over the other salts was not as pronounced.

DISCUSSION

PREPARATION OF DETERGENT MIXTURE——The method of incorporating the magnesium ion into the composition seemed to be of little significance. It was accomplished by—

(1) Partial neutralization of the sulfonic acid with magnesium oxide;
(2) Adding magnesium sulfate to the neutralized slurry;
(3) Intimate dry blending; and
(4) Adding magnesium sulfate to the dilute detergent solution prior to the wash test.

PREPARATION OF SOIL CLOTH——Inasmuch as no commercial standard wool soil cloth was available at the time of the experiments, a synthetic soil was prepared according to the method of Price *(5)*. The preparation consisted of 50 g of anhydrous lanolin and 50 g of white mineral oil melted together. Six grams of "Pure Germantown Lamp Black" (Felton Sibley & Co, Phila, Pa) were mixed in uniformly and then the mixture was dispersed in one gallon of solvent (Stoddard solvent, carbon tetrachloride, etc). Swatches, 3 by 4 inches, cut from 8 oz, 54-inch, white virgin wool flannel were impregnated with 3 cc of the soil solution and air dried. The best method found for obtaining uniform dispersion of the soil on each swatch was to place 3 cc of the soil solution along with one swatch in an 8 oz, wide-mouth glass jar and shake until the soil solution was entirely absorbed by the swatch. The soil swatch was then removed and air dried for 24 hours. These were used for washing tests.

WASHING TESTS——The detergency values were obtained by slightly modifying the standard Launder-Ometer tests. A soil swatch was added to a jar containing 300 cc of ¼% of the compound to be tested and twenty ¼-inch stainless steel balls. The jars were rotated in a constant temperature bath at 50° C for thirty minutes. At that time the swatch was removed, squeezed out and rinsed with clear water. If the solution showed detergency, another swatch was placed in the jar and the test repeated. This operation was continued until the solution had lost all its detergent properties. The swatches were dried and the extent of soil removal determined by measuring the reflectance of each swatch with a Hunter Reflectometer. The soil removal capacity for a wash solution was obtained by adding the readings of each swatch and subtracting eleven (the reading for the unwashed soil cloth). This value is the "reflectance index" in the tables in this report.

RESULTS

The experimental values obtained from the test on wool wherein magnesium sulfate was used to aid the cleansing power of a mixture of sodium sulfate and sodium dodecyltoluenesulfonate are summarized in accompanying tables.

In Tables I and II, the effect of varying the ratios of magnesium to sodium on removing soil is shown. Since approximately 40% organic concentration of the sodium alkylarylsulfonate is generally acceptable as a desirable concentration, that value is kept constant in all these considerations. It appears that the optimum ratio of magnesium sulfate, sodium sul-

TABLE I

EFFECTS OF INCREASING THE RATIO OF $MgSO_4$ TO ORGANIC MATERIAL*

(Sodium dodecyltoluenesulfonate** — abbreviated SDTS — 0.25% of 40% SDTS and 60% Na_2SO_4 were used and $MgSO_4$ was added to that solution.)

Mole Ratio of $MgSO_4$ to SDTS	*Mole Ratio of $MgSO_4$ to Na_2SO_4*	*Reflectance Index of the Washed Wool Swatches*
No Mg	No Mg	12
0.25	0.08	28
0.5	0.17	57
0.75	0.25	79
1.0	0.27	86
1.25	0.33	109
1.5	0.39	115
2.0	0.52	110
2.5	0.65	101
3.0	0.78	81
3.5	0.91	49

* In all these tests distilled water was used.
** Sodium dodecyltoluenesulfonate was prepared by sulfonating commercial dodecyltoluene purchased from the Sharples Continental Corp.

TABLE II

EFFECTS OF VARIOUS PERCENTAGES OF $MgSO_4$ AND Na_2SO_4

(Sodium dodecyltoluenesulfonate content was kept at 40% in all tests.)

Composition of Inorganic Material		Mole Ratio:	Reflectance Index of the
% $MgSO_4$	% Na_2SO_4	$MgSO_4/Na_2SO_4$	Washed Wool Swatches
10	50	0.24	103
15	45	0.39	135
20	40	0.59	130
25	35	0.84	117
30	30	1.18	36
35	25	1.65	13
40	20	2.36	11
45	15	3.50	10
50	10	5.90	7

fate and sodium dodecyltoluenesulfonate is approximately 15:45:40, respectively, expressed as weight percentage of dried product.

The capacity of this detergent composition was found to be almost linear as is shown from the data in Table III.

The effects of other salts are shown in Tables IV and V. Calcium improves the detergency of sodium dodecyltoluenesulfonate, but to a much smaller extent than magnesium. A comparison of Tables I and II with Table IV clearly shows this. The calcium is about one-third as efficient as magnesium. A number of other salts including aluminum, zinc, cadmium, boron, and lithium were tried and, while some showed an improvement, none were efficient enough for practical consideration.

Detergents prepared from keryl benzene, dodecylbenzene, and nonylnaphthalene were all improved by inclusion of magnesium sulfate in the detergent composition, but the improvement was much less than in the case of dodecyltoluene. This is clearly shown in Tables VI, VII and VIII. This activator effect of magnesium sulfate is still pronounced in acid solutions and is effective down to a pH of 3.

TABLE III

CAPACITY OF THE DETERGENT OF COMPOSITION: 40% SDTS, 45% Na_2SO_4, 15% $MgSO_4$

Conc of Detergent	Reflectance Index	No Swatches Washed
0.1%	9	1
0.2	84	3
0.3	184	6
0.4	290	8
0.5	394	10
0.7	648	16

TABLE IV

EFFECTS OF CALCIUM ON THE DETERGENCY OF SODIUM DODECYLTOLUENESULFONATE AND Na_2SO_4

[$Ca(OH)_2$ was added to a 0.25% solution of SDTS (40%) and Na_2SO_4 (60%)]

Conc $Ca(OH)_2$ %	Mole Ratio of $Ca(OH)_2$ to SDTS	Reflectance Index
0.00	....	9.3
0.002	0.098	9.9
0.004	0.19	10.8
0.006	0.28	15.2
0.008	0.39	17.4
0.010	0.49	27.0
0.015	0.73	36.5
0.020	0.98	28.8
0.025	1.22	26.0
0.030	1.46	13.9

TABLE V

EFFECTS OF OTHER METAL SALTS ON SODIUM DODECYLTOLUENESULFONATE

(The salts were added in a 1:1 mole ratio of added salt to SDTS in a 0.25% solution of the detergent consisting of 40% SDTS and 60% Na_2SO_4.)

Salt Added	Reflectance Index
$AlCl_3$	0.0
$ZnCl_2$	88
$CaSO_4$	25
$CdSO_4$	68
$LiCl$	9
$Na_2B_4O_7$ adjusted to pH = 9.2	36

TABLE VI

EFFECT OF MAGNESIUM SULFATE ON OTHER DETERGENTS

(The detergent solution was made up as 0.25% concentration and the $MgSO_4$ added as indicated.)

Detergent	$MgSO_4$ %	Reflectance Index
Sodium nonylnaphthalenesulfonate	None	2.1
Sodium nonylnaphthalenesulfonate	0.03	26
Sodium alkylarylsulfonate	None	2.6
Sodium alkylarylsulfonate	0.03	17.2
SDTS 40%, Na_2SO_4 60%	0.03	87
SDTS 40%, Na_2SO_4 60% adjusted to pH 9	0.03	101

This effect of magnesium may be an explanation as to why it is occasionally mentioned that some detergents are more effective in hard water than in soft water.

SUMMARY

Magnesium ion has been found to increase several fold the detergency properties for wool of sodium dodecyltoluenesulfonate mixed with sodium sulfate. Approximately 40% organic content, 15% magnesium sulfate, and 45% sodium sulfate seems to be optimum. Other sulfonates and ions were tested and found to be inferior to the magnesium dodecyltoluenesulfonate combination.

TABLE VII

EFFECT OF VARIOUS CONCENTRATIONS OF $MgSO_4$ ON SODIUM NONYLNAPHTHALENE SULFONATE

[0.25% solution of the detergent (40% sodium nonylnaphthalenesulfonate and 60% Na_2SO_4) was made up and the $MgSO_4$ was added to this as shown below.]

Mole Ratio of $MgSO_4$ to Sodium Nonylnaphthalene-sulfonate 40% Mixture	Reflectance Index
No $MgSO_4$	2
0.25	11
0.5	25
0.75	31
1.0	34
1.5	35
2.0	23
2.5	17
3.0	12

TABLE VIII

EFFECTS OF VARIOUS CONCENTRATIONS OF $MgSO_4$ ON DODECYLBENZENESULFONATE

(This is a duplicate of Table VI except the detergent contained 40% sodium dodecylbenzenesulfonate instead of 40% sodium nonylnaphthalenesulfonate.)

Mole Ratio of $MgSO_4$ to Sodium Dodecylbenzene-sulfonate	Reflectance Index
0.25	4.5
0.5	5.2
0.75	9.8
1.0	18
1.5	34
2.0	32
2.5	21
3.0	22

TABLE IX

EFFECT OF pH ON THE MAGNESIUM SULFATE BUILT DETERGENT

(0.25% solution of the detergent: SDTS 40% and Na_2SO_4 60%. To this $MgSO_4$ was added.)

Mole Ratio of $MgSO_4$ to SDTS	pH	Reflectance Index
No $MgSO_4$	7.0	10
No $MgSO_4$	5.0	7
No $MgSO_4$	3.0	5
1	7.0	73
1	5.0	67
1	3.0	49

REFERENCES

1) German Patent 690,628 (April 11, 1944).
2) Harris, *Rayon Textile Monthly* 26, 77-80, 142-144 (1945).
3) Harris, U S Patent 2,298,696 (Oct 13, 1943).
4) Miles and Ross, *Ind Eng Chem* 35, 1221-4 (1943).
5) Price, et al "Surface Active Agents", *Annals of the New York Academy of Science* XLVI Art 6, 347-530, published by the Academy 3/15/46.
6) Zizinia and McKenna, U S Patent 2,364,767 (Dec 12, 1944).

• PATENT DIGEST •

PAUL WENGRAF

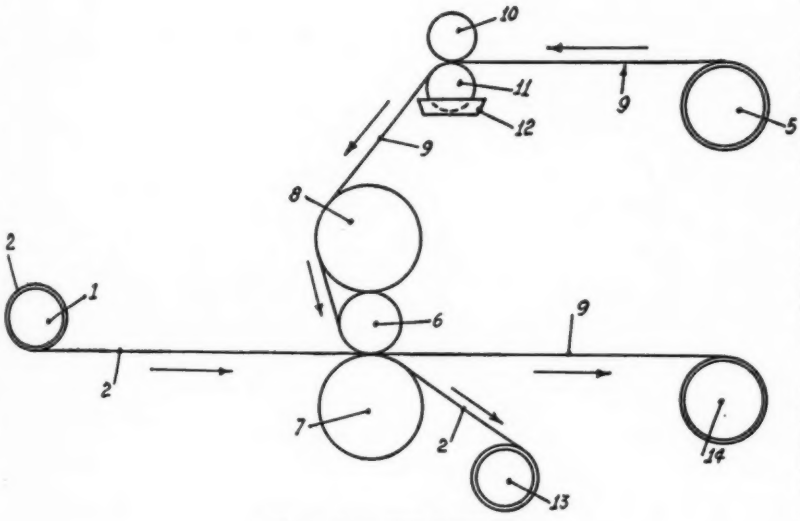


Figure 1 — USP 2,583,286

PRINTING TEXTILES—— Colored Patterns Transferred from Printed Paper Sheets D, 1

U S Pat 2,583,286
(Albini——Colombo——Jan 22, 1952)

This patent protects a printing method similar in some respect to the Tschechonin ("Orbis") process or to that described in Brit P 592,484 (cf *Am Dyestuff Reptr* 37, 656 (1948)) in that a pre-formed colored pattern is transferred by contact to a moistened textile fabric.

A strong paper strip is first coated with a thick paste prepared from starch, acetic acid and formaldehyde, then dried and further coated with a varnish-like mass of zinc stearate, oleic resin and a solvent, such as turpentine. These two coatings are designed to prevent the paper from absorbing the printing ink and to make it easily removable in the subsequent printing operation. The printing ink may contain acid, direct or basic colors, or mineral pigments (Prussian blue, manganese brown, chromium yellow) incorporated into a varnish-type thickener, which is described in detail in the specification. The ink also contains linseed oil, turpentine, zinc stearate and colophony emulsified in a dextrine thickener.

The prepared paper is printed according to one of the standard paper-printing methods (lithography, typography, etc). Printed sheets (3)—see Fig 2—are fixed on a strong web of paper (2) which is fed from a roll (1) to a three-roll calender (see Fig 1). The fabric (9) to be printed comes from (5), passing a pair of moistening rollers (10 and 11). The pattern printed on (2) is transferred at the nip between rolls (6 and 7) to the fabric (9), which can be further subjected to necessary fixing operations.

There is no special fixing process within the scope of this invention, but it may be assumed that mineral pigments are fixed by applying suitable emulsions and subsequent heating and steaming.

An advantage of this method, as pointed out by the inventor, is that it makes possible reproductions of "miniatures, colored images, wood engravings and the like".

Among other references cited by the Patent Office:

U S Pat 2,353,717 (Sylvania Ind Corp/1944): transferring a film to an article for the purpose of decorating by applying a layer of a thermoplastic material, such as a polyvinyl resin, to a smooth backing of regenerated cellulose sheet, tin foil or the like.

U S Pat 2,290,365 (Wynne/1942): an apparatus for transferring decalcomanias from their normal backing to glass or other surfaces by mechanical means, continuously operating and wetting the sheets, in which manner the backing support is detached and wound on a cylinder.

U S Pat 1,820,559 (Colorfuse Ltd/1931): transferring a design by producing it with ink on a temporary support coated with a fusible wax-resin preparation or the like, forming an emulsion with the printing ink and transferring it to fibrous material by vapor and pressure.

U S Pat 1,810,294 (Parks & Woolson Co/1931): a machine for applying decalcomanias on cloth at regular intervals by providing means for advancing or adjusting the carrier tape, on which stamps are placed while the device is in operation.

Further reference is made to U S Pat 2,050,260 (Black) and U S Pat 1,993,524 (Decorative Development) in which various colors from a nonabsorbent and resilient sheet are transferred; this sheet and the fabric are joined under pressure in the presence of a solvent.

(Parallel Brit P 670,174 of April 16, 1952).

Figure 2 — USP 2,583,286

SHRINKPROOFING WOOL—— Permanganate Treatment at Elevated Temperature Under Pressure A, 2, 04

U S Pat 2,548,774
(Coe, Jr——April 10, 1951)

In the introduction to this patent, the inventor states that dilute permanganate solutions can prevent wool shrinkage only slightly. As early as 1907 it was proposed by Kammerer (see references below) that free permanganic acid be used, but it has been observed since that any treatment at temperatures below 100° C has the drawbacks of penetrating the wool substance poorly, affecting only the surface and thereby unfavorably changing the natural feel or hand of the material.

In contrast to former methods, this

current invention comprises a permanganate shrinkproofing treatment at elevated temperatures and superatmospheric pressure. In this case wool undergoes hydrolyzation at the cystine linkages, liberating H_2S, which is partly oxydized by permanganate to H_2SO_3 and H_2SO_4. The three acids react with potassium permanganate to produce permanganic acid. Thus a wool fiber impregnated with permanganate and subjected to pressure hydrolyzation will react with the readily oxidizing permanganic acid, replacing the unstable sulfhydryl linkages by stable bonds, such as R-S-R', R-S-NH-R' and other chains, and result in a permanent set of the wool substance. This oxidizing effect advantageously occurs only when $KMnO_4$ comes in contact with the wool, thereby producing a smooth effect. A further advantage is a simultaneous bleaching and shrinkproofing reac ion under essentially neutral conditions.

Example: virgin wool is treated with a solution of 250 g potassium permanganate in 2 liters of water in an autoclave at 15 lbs pressure and temperatures over 100° C for five minutes. Thereafter the brown color of manganese compounds is removed with diluted solutions of sodium bisulfite and the material finally neutralized with Na_2CO_3. The wool is reportedly found to be absolutely shrinkproof compared with nontreated wool, which shrank about 25%.

Among other references cited by the Patent Office:

Brit P 5612/1907 (Kammerer): shrinkproofing wool by a treatment with sulfuric acid and permanganate, the acid amount being sufficient to prevent a deposit of MnO_2. Even penetration is claimed.

Brit P 569,730/1944 (Stevenson Dyers): a combined hypochlorite-permanganate shrinkproofing treatment; p-toluene sulfonchloramide may be used as an option to hypochlorite.

Brit P 586,020/1947 (Wolsey Ltd): immersing wool to be shrinkproofed in acid permanganate solutions at a pH below 2.0 for a time not exceeding a few minutes.

Brit P 603,379/1948 (Vicara & Co): immersing 100 lbs of wool for 30 minutes in a 250-gallon bath containing 4 lbs $KMnO_4$ and 7.7 lbs H_2SO_4 66%, and removing MnO_2 by reduction or acidification.

DYEING FUR SKINS—— Metallized Azodyes in Solvent Solutions C, 4, 06

U S Pat 2,585,610
(Ciba Ltd——Wittwer——Feb 12, 1952)

Normal wool dyeing operations requiring elevated temperatures cannot be used for dyeing fur skins, mainly because of the sensitivity of the material under these conditions.

It has been found that fur skins and other animal fibers can be dyed well at temperatures between 40-60° C in an aqueous-solvent mixed dyebath containing from 20-80% of an aliphatic water miscible alcohol (methanol, ethanol, propanol) low glycols or low esters (ethylacetate).

The dyestuffs employed in this process are particularly complex metal compounds of azodyes as mentioned in the title and in the claims. Acid or basic dyes that have no free sulfonic or carboxylic groups and are only slightly soluble in water may also be used. The addition of a volatile acid (formic or acetic) in an amount of 5 g per liter of dyebath is said to be advantageous. It is reported that very uniform dyeings of good penetration and reduced shrinkage result. Some auxiliaries of the "Ultravone" type help to counteract the shrinking tendency of the skin portion, which is usually dyed in a lighter shade than the hair portion.

Example: tanned sheepskin is dyed in a bath of equal parts of water and alcohol, containing as a dyestuff the complex cobalt compound of a dye obtained from diazotized nitroaminophenol and phenyl methyl pyrazolone plus a small amount of formic acid. The goods are immersed at 30° C, which temperature is raised within 30 minutes to 50-55° C. Dyeing continues for another half hour at this temperature. The hair is dyed a bright orange, the skin itself remains almost uncolored.

Among other references cited by the Patent Office:

U S Pat 2,374,106 (Du Pont /1945): dyeing nylon with water-dispersed water-insoluble complexes of monoazodyes, e g, Cu-, Cr-, Co-, Ni- or Mn complexes.

U S Pat 2,292,433 (Celanese/1942): wool-cellulose acetate mixtures dyed with acid dyes in a bath containing swelling agents, such as diluted alcohol, whereby the wool part remains undyed while the cellulose acetate adsorbs the acid dye.

U S Pat 2,163,043 (Kritchewsky-Rit Products/1939): a hair dyeing composition containing urea or urea derivatives as assistants.

U S Pat 1,186,412 (Gaudit/1932): dyeing or printing with acid dyes containing a very high percentage of formic acid. This is said to result in sufficient fixation at room temperature.

Further reference is made to Gaudit's U S Pat 2,029,568 (1936), which discusses a printing process that consists of adding glycerine and/or water soluble alcohols to metal complex azodyes thickened as usual, printing with the resulting paste and steaming.

DYEING OR PRINTING LAKES of Basic Dyes and Phosphotungstic or Phosphomolybdic Acids C, 4, 01

U S Pat 2,589,953
(Calico Printers Ass'n——Miller——
March 18, 1952)

Basic dyes are known to form lakes of high light fastness by precipitation with phosphomolybdic or phosphotungstic acids. This property has been used for preparing the brilliant and (at the same time) lightfast "Fanal Dyes". One of the pertinent patents is U S Pat 1,912,878 of I G Farbenindustrie A G.

This type of dye however could only be used hitherto in the form of insoluble lakes fixed on the fiber by means of an adhesive, thus lacking fastness to crocking.

A perfect solution to the problem of coloring fibers with the aforementioned dyestuff compositions could be seen only in the formation of lakes in situ within the fiber. The inventor developed a practical method of applying the dyestuff and the mordant (i e, the phosphomolybdic or tungstic acid) in a mixture, at the same time avoiding precipitation, and finally forming the lake by an acid or acid-steam aftertreatment.

If basic dyes are dissolved in water (with an optional addition of lower alcohols or glycols and an admixture of alkali salt of one of the above acids), precipitation is prevented by keeping the solution at a pH between 5 and 12.5. Lake formation occurs immediately at a pH below 5, while on the other hand strongly alkaline media with pH values above 12.5 are apt to destroy the basic dyes and give poor shades. Buffering salts, e g, sodium phosphate, citrates or borates, may be used to maintain the solution at the pH value required in this operation.

The inventor found that the insoluble lakes appear to be precipitated within the fiber rather than on the surface or between the elemental fibers, thereby improving the fastness to crocking considerably. Phosphomolybdic acid —— $H_7[P(Mo_2O_7)_6]$——used in the form of its alkali salt is preferred as the lake-forming acid. It may also be generated in situ from sodium molybdate plus phosphoric acid (or alkaliphosphate). In the latter event alkaliphosphate acts as a buffering substance.

Example: 1 g Victoria Blue is dissolved in 8 g ethanol plus 30 g water and urea (as a hygroscopic agent). This solution is stirred into a carob bean thickener. 3 g sodium phosphomolybdate are added and the paste made up to 100 g. After the printing operation the insoluble lake is precipitated by an acid steam passage.

(Concluded on Page 435)

Proceedings of the
American Association of Textile Chemists and Colorists

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July 7, 1952

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CALENDAR

COUNCIL

September 12 (Philadelphia); November 6 (Boston)

GENERAL RESEARCH COMMITTEE

(See Council dates)

NATIONAL CONVENTIONS

November 6-8, 1952 (Hotel Statler, Boston); September 17-19, 1953 (Hotel Conrad Hilton, Chicago); 1954 (Atlanta, Ga).

MID-WEST SECTION

October 4 (Hotel Bismarck, Chicago).

NEW YORK SECTION

September 26, October 24, November 21 (Kohler's Swiss Chalet, Rochelle Park, N J); January 30, February 27 (Hotel Statler, New York, N Y); April 17, May 22 (Swiss Chalet)

NORTHERN NEW ENGLAND SECTION

December 6 (Annual Meeting, Lowell Textile Institute).

PIEDMONT SECTION

Sept 13 (Hotel Charlotte, Charlotte, N C).

RHODE ISLAND SECTION

Oct 24 (Providence Engineering Society); December 5 (Annual Meeting, Sheraton-Biltmore, Providence, R I).

SOUTH CENTRAL SECTION

Aug 22-23 (Outing, Chattanooga Golf & Country Club); December 6 (Patten Hotel).

SOUTHEASTERN SECTION

Sept 13 (Biltmore Hotel, Atlanta).

WESTERN NEW ENGLAND SECTION

Oct 3, Nov 14 and Dec 12 (Rapp's Restaurant, Shelton, Conn).

LAPEL BUTTONS FOR MEMBERS

These buttons bear the seal of the AATCC and may be purchased by members in good standing from the National Secretary at $2.00 each.

DR HAROLD C CHAPIN
Lowell Textile Institute
Lowell, Massachusetts

OTHER EVENTS

AMERICAN CHEMICAL SOCIETY

7th National Chemical Exposition, Sept 9-13, Chicago Coliseum, Chicago, Ill. This will include the 2nd International Art Exhibit.

AMERICAN OIL CHEMISTS SOCIETY

Short Course industrial trips, July 8-9, 10-11.

AMERICAN SOCIETY FOR TESTING MATERIALS

1953 Annual Meeting, June 29-July 3, Chalfonte-Haddon Hall, Atlantic City, N J.

AMERICAN STANDARDS ASSOCIATION

3rd National Standardization Conference, Sept 8-10, Museum of Science and Industry, Chicago, Ill.

34th Annual Meeting, November 19, Waldorf-Astoria, New York, N Y.

AMERICAN TEXTILE MACHINERY ASSOCIATION

Exhibit of American Textile Machinery, April 26-May 1, 1954, Atlantic City Exhibition Hall, Atlantic City, N J.

CANADIAN TEXTILE SEMINAR

September 11-13, Queen's University, Kingston, Ont.

FIBER SOCIETY

Sept 10-11 (Princeton, N J).

INTERNATIONAL COTTON AND RAYON EXHIBITION

Sept 27-Oct 12, Busto Arsizio, Italy.

LEIPZIG FAIR

September 7-17, Leipzig, Germany.

NATIONAL ASSOCIATION OF HOSIERY MANUFACTURERS

41st Knitting Arts Exhibition, April 27-May 1, 1953, Atlantic City Auditorium, Atlantic City, N J.

NEW YORK BOARD OF TRADE (DCAT)

26th Annual Meeting: September 25-28, Pocono Manor Inn, Pocono Manor, Pa.

27th Annual Drug, Chemical and Allied Trades Dinner, March 5 (1953), Waldorf-Astoria, New York, N Y.

TEXTILE RESEARCH INSTITUTE

23rd Annual Meeting, Nov. 17-18, Hotel Commodore, New York, N. Y.

TEXTILE WORKSHOP

June 16-July 25, Lowell Textile Institute, Lowell, Mass.

1952 NATIONAL CONVENTION

TEXTILE DYEING & FINISHING EXPOSITION

HOTEL STATLER

BOSTON, MASSACHUSETTS

NOVEMBER 6, 7, 8, 1952

America Advances Through Creative Chemistry

Report of a Research Committee——

INSTRUMENTS FOR MEASURING CREASE RESISTANCE

AATCC RESEARCH COMMITTEE FOR EVALUATION OF DURABLE FINISHES

AS part of the program of the American Association of Textile Chemists and Colorists Committee on Evaluation of Durable Finishes an interlaboratory comparison has been completed with the American Cyanamid Company's Roller Pressure Tester (RPT) *(1)* and Monsanto Chemical Company's Wrinkle Recovery Tester (WRT) *(2)* to measure the crease resistance of selected fabrics. Both instruments have been designed to correct recognized deficiencies of the Tootal Broadhurst Lee (TBL) *(3) method* generally used in industrial evaluation of crease resistance; so it was the objective of this comparison to determine if one or both might be suitable for an AATCC standard method of evaluation of crease resistance.

COMMITTEE

Martin H Gurley, Jr, *Chairman*

K H Barnard — D Burkes
A I Anderson — E R Kaswell
P J Ariente — R F Nickerson
G L Baxter — A D Nute
J H Brant — R A Pingree

Two instruments for evaluating the crease resistance of fabrics became available in addition to the Tootal-Broadhurst Lee method generally used.

A project was assigned to the Durable-Finishes Committee to compare the Monsanto Company's Wrinkle Recovery Tester and the American Cyanamid Company's Roller Pressure Crease Recovery Tester. Under the chairmanship of Martin Gurley, Jr, the Committee studied the two instruments, made interlaboratory tests and present the results to encourage further evaluation on a broader scale. In accordance with this objective, the General Research Committee accepted both instruments as Tentative Standard.

In addition to other active projects, the committee, under the present chairman, Alden D Nute, continues interest in crease-testing methods, welcoming comments and further evaluation.

H W STIEGLER
Director of Research

RESULTS

In this comparison it was found that

1) In the larger number of tests there are significant differences between the results secured with the two instruments for the same fabrics in the same laboratory.

2) In most of the results that were statistically analyzed, significant differences were found between the results secured in different laboratories on the same fabric and same RPT but not necessarily the same WRT.

3) In most cases analyzed the RPT showed greater sensitivity than did the WRT when sensitivity is defined as the increase in crease-recovery angle developed by treatment of the fabric.

4) In the majority of results the RPT showed a lower crease-recovery angle than did the WRT.

5) Analysis of variance in results from two co-operating laboratories indicates the major portion of determinable variance to be due to the operators.

6) The reproducibility of results on the same fabrics in several WRT's justifies the use of several WRT's to save time in commercial testing.

7) A study of the effects of sample preparation by dinking (cutting with a die), cutting with scissors and cutting with razors indicated that the latter two are generally satisfactory, but dinking is best.

8) Both instruments are readily explained to laboratory technicians in a few minutes, and operators can be classed as experienced after about two hours of work with either device.

9) Both instruments were shown by these tests to be more discriminatory at the extremes of angular recovery than in the middle range.

10) Variance attributable to differences between warp and filling in these fabrics is so much less than that between operators that it is suggested that for control purposes measurements can be made in one direction only.

CONCLUSION

Inasmuch as both instruments give results that are significantly different but which correlate with fabric appearance, it is recommended that both instruments be submitted to the Research Committee membership for approval as tentative means of measuring changes in crease-recovery angles of fabrics until such time as sufficient correlation with actual use of crease-resistant fabrics justifies advancing either or both instruments to standard, or until a new instrument with greater utility is developed.

DETAILED DISCUSSION

INSTRUMENTS—Prior to the development of these two instruments the crease resistance of fabrics was probably most frequently measured by the TBL test as developed by Tootal Broadhurst Lee Company *(3)*.

The TBL test, widely used to measure crease resistance of fabrics, consists of folding a small (2 x 4-cm) specimen and compressing it under a specified weight for a

TABLE I

COMPARISON OF REPRODUCIBILITY AMONG THREE MONSANTO TESTERS

Sample	*Direction of Test*	*WRT*	*Recovery Angle (°)*	*SD (°)*	*Coeff of Variation (%)*	*Standard Error (°)*	*F Test*
B t	Warp	#1	137.9	2.39	1.7	0.69	Critical F = 3.30 at 5% Sig level; F determined = 0.865. ∴ No significant difference
	Warp	#2	140.0	6.89	4.92	1.99	
	Warp	#3	137.6	5.376	3.91	1.55	
C u	Fill	#1	101.3	5.61	5.5	1.62	Critical F = 3.30; F determined = 1.03. ∴ No Sig Diff.
	Fill	#2	98.6	3.77	3.82	1.08	
	Fill	#3	100.1	3.796	3.79	1.09	
D u	Warp	#1	79.6	5.12	6.43	1.47	Critical F = 3.30; F determined = 1.81. ∴ No Sig Diff.
	Warp	#2	82.6	9.69	11.7	2.80	
	Warp	#3	85.16	4.93	5.78	1.27	

Note: dink cut used exclusively.

fixed time and then removing the load and dropping the sample onto a horizontal wire so that the sample is suspended at the fold. After the sample has recovered for a fixed time, the distance across the bottom of the inverted "V" is measured in centimeters as the anticrease or crease-resistant value.

The TBL method has been criticized as defective in that

1) As the angle of the recovered crease increases, the change in the chord length (the distance between the ends of the inverted "V") decreases markedly, and this results in poor discrimination between samples of high crease resistance.

2) As the softness and the crease resistance of fabrics increase, the effect of gravity is more marked on the ends of the sample and lowered anticrease values are obtained.

3) Twisting of certain samples made it difficult for the technician to determine accurately the chord length, particularly in samples of unbalanced fabrics.

4) Thickness and compressibility of fabrics markedly affect the TBL test results.

In order to overcome these deficiencies, the RPT and WRT instruments were developed. Both have been designed to measure the angle of crease at the apex or very close thereto. This minimizes the effects of gravity and twisting while increasing the sensitivity at high values of crease resistance.

Both instruments during the course of their development were compared with the TBL method, and each has been shown to be more sensitive than the TBL, particularly with markedly crease-resistant fabrics.

Each instrument as used in this comparison applies a prescribed pressure for a fixed time to the same-sized specimen and permits recovery after removal of the pressure for a fixed time before reading of the crease-recovery angle. In the RPT the pressure is two kilograms and the time for any single area of the sample is very small, both factors being instrument constants. The recovery time and angle measurement are subject to operator errors. The WRT method uses a one-and-one-half-pound weight applied to the folded specimen in the tester clamp for a five-minute period. The specimen is then unloaded and transferred to the WRT proper, where it is allowed to recover for five minutes before the angle of recovered fabric is measured. In this method the times of pressing and recovery as well as measurement of angle are subject to operator errors. In this comparison all times and pressures were as specified in Monsanto Chemical Company Technical Bulletin No. T-7 of 1 December 1947, which describes the instrument and its operation.

For this comparison only one RPT (model 2) was available, and it was sent in turn to each co-operating laboratory. Those laboratories which did not have WRT's were supplied with instruments from the manufacturer's stock. In several laboratories a number of WRT's were used as in

TABLE II

DESCRIPTIONS OF FABRICS USED IN COMPARISON OF MONSANTO WRT AND CYANAMID RPT

Identification	Type	Oz/sq yd	Ends/in	Picks/in
A	100% wool worsted	9	70	42
B u	Rayon/wool Gabardine 60/40	8.8	60	48
B t	Same, treated	9.2	64	50
C u	100% spun-rayon	4	80	44
C t	Same, treated	4.4	74	44
D u	Cotton	3	92	72
D t	Same, treated	3.4	100	64

TABLE III

EFFECT OF TYPE OF CUT (DINK, SCISSORS, RAZOR) ON RECOVERY ANGLE

Sample	Test Direction	Cut	Crease Angle Recovery WRT #1	RPT	Std Dev (°)	Coeff Var (%)	Std Error	F Test
C u	Fill	dink	101.3		5.61	5.50	0.75	F=.05. F=3.30
	Fill	scissors	100.6		6.55	6.51	1.89	at 5% level.
	Fill	razor	100.9		3.73	3.69	1.08	∴ Not sig
C u	Fill	dink		97.9	2.60	2.65	0.75	F=1.17. F=3.30
	Fill	scissors		95.6	4.72	4.93	1.36	at 5% level.
	Fill	razor		95.6	5.95	6.22	1.71	∴ Not sig
D u	Warp	dink	79.6		5.12	6.43	1.47	F=0.45. F=3.30
	Warp	scissors	81.		5.597	6.9	1.62	at 5% level.
	Warp	razor	82.3		7.24	8.79	2.09	∴ Not sig
D u	Warp	dink		59.5	11.02	18.52	3.18	F=0.16. F=3.30
	Warp	scissors		55.3	4.42	7.99	1.62	at 5% level.
	Warp	razor		59.5	10.73	18.03	2.09	∴ Not sig

TABLE IV

RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "A"

Sample	Direction of Test	Recovery Angle WRT (°)	Rank	RPT (°)	Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	% Coeff of Var WRT	% Coeff of Var RPT	Std Error WRT (°)	Std Error RPT (°)	T-Test of Sig†	Sig Diff*
A	Warp	154	2	154	1.5	0	1.73	2.83	1.12	1.84	0.77	1.26	0.24	No
	Fill	156	1	154	1.5	— 2	2.45	2.61	1.57	1.69	1.09	1.17	0.68	No
B u	Warp	135	8	139	5	+ 4	5.15	3.55	3.81	2.55	2.30	1.58	1.27	No
	Fill	136	7	134	6	— 2	1.26	6.14	0.93	4.58	0.56	2.74	0.70	No
B t	Warp	147	4	151	3.5	+ 4	2.05	0.63	1.39	0.42	0.92	0.28	3.16	Yes
	Fill	143	6	151	3.5	+ 8	2.65	1.95	1.85	1.29	1.18	0.87	4.88	Yes
C u	Warp	106	11.5	95	11	—11	7.55	8.15	7.12	8.58	3.37	3.64	2.01	No
	Fill	112	10	93	12	—19	4.15	2.10	3.71	2.26	1.85	0.94	8.96	Yes
C t	Warp	146	5	131	7	—15	3.03	3.58	2.08	2.73	1.35	1.60	6.78	Yes
	Fill	151	3	130	8	—21	2.79	4.88	1.85	3.75	1.25	2.18	7.33	Yes
D u‡	Warp	79	13	69	13	—10	14.72	4.84	18.63	7.01	6.57	2.16	1.27	No
	Fill	78	14	67	14	—11	6.43	2.19	8.24	3.27	2.87	0.98	3.27	Yes
D t‡	Warp	106	11.5	106	9	0	5.31	5.27	5.01	4.97	2.37	2.35	0.11	No
	Fill	114	9	104	10	—10	5.55	0.88	4.87	0.85	2.48	0.39	3.86	Yes

* Probability that WRT and RPT crease angles are the same is high for "No", low for "Yes".
† Critical value of T at the 5% level is 2.306.
‡ Untreated and treated tickets appeared reversed; results as samples should have been marked.
Note: five specimens were tested from each sample.

TABLE V

RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "B"

Sample	Direction of Test	Recovery Angle WRT (°)	WRT Rank	Recovery Angle RPT (°)	RPT Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	% Coeff of Var WRT	% Coeff of Var RPT	Std Error WRT (°)	Std Error RPT (°)	T-Test of Sig*	Sig Diff†
A	Warp	150	2	136	2	—14	2.76	1.90	1.84	1.40	1.23	0.69	7.99	Yes
	Fill	154	1	140	1	—14	0.63	4.56	0.41	3.27	0.28	2.04	5.81	Yes
B u	Warp	131	5	123	6	— 8	2.61	2.10	1.99	1.70	1.17	0.94	5.34	Yes
	Fill	123	6	121	8	— 2	1.26	2.57	1.02	2.12	0.56	1.15	1.54	No
B t	Warp	132	4	130	3	— 2	2.72	1.55	2.06	1.19	1.21	0.69	1.52	No
	Fill	143	3	129	4	—14	2.19	2.76	1.53	2.14	0.98	1.23	2.46	Yes
C u	Warp	89	11	79	11	—10	2.79	3.13	3.13	3.96	1.25	1.40	4.77	Yes
	Fill	88	12	69	12	—19	1.67	1.55	1.90	2.25	0.75	0.69	16.50	Yes
C t	Warp	119	7	122	7	3	2.05	1.79	1.72	1.47	0.92	0.80	2.67	Yes
	Fill	115	8	126	5	11	2.10	1.73	1.82	1.37	0.94	0.77	8.27	Yes
D u‡	Warp	72	14	52	14	—20	1.73	1.41	2.40	2.72	0.77	0.63	18.88	Yes
	Fill	82	13	54	13	—28	3.16	3.03	3.85	5.61	1.41	1.35	13.00	Yes
D t‡	Warp	102	9	89	10	—13	2.86	5.66	2.81	6.36	1.28	2.53	4.10	Yes
	Fill	101	10	104	9	3	4.96	1.95	4.91	1.88	2.21	0.87	0.91	No

* Critical value for T at the 5% level is 2.306.
† Probability that WRT and RPT crease angles are the same, is high for "No", low for "Yes".
‡ Results as samples should have been marked, not as marked when received.
Note: five specimens were tested from each sample.

TABLE VI

RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "C"

Sample	Direction of Test	Recovery Angle WRT (°)	WRT Rank	Recovery Angle RPT (°)	RPT Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	% Coeff of Var WRT	% Coeff of Var RPT	Sig Diff*
A	Warp	141	4	144	6	3	3	3	2.0	2.4	!
	Fill	149	1	147	2.7	— 2	4	4	2.7	2.7	No
B u	Warp	135	7.5	131	7	— 4	2	2	1.8	1.7	Yes
	Fill	135	7.5	128	8	— 7	3	3	2.0	2.3	Yes
B t	Warp	143	3	150	1	7	2	3	1.4	2.2	Yes
	Fill	145	2	146	5	1	3	2	1.8	1.7	No
C u	Warp	95	12	89	12	— 6	7	6	7.0	7.0	No
	Fill	106	10	92	11	—14	7	4	6.0	5.0	Yes
C t	Warp	137	6	147	2.7	10	5	2	3.6	1.7	Yes
	Fill	138	5	147	2.7	9	5	4	3.3	2.9	Yes
D u	Warp	77	14	73	14	— 4	7	4	9.0	5.0	No
	Fill	86	13	80	13	— 6	6	3	7.0	4.0	Yes
D t	Warp	100	11	102	10	2	4	3	4.0	2.9	No
	Fill	111	9	108	9	— 3	4	4	3.6	4.1	No

* Probability that WRT and RPT crease angles are the same, is high for "No", low for "Yes".

commercial testing to reduce the time.

Table I presents results which indicate that there is no significant difference between three WRT's selected at random. These results show the reproducibility of results when several WRT's are used to save time in commercial testing.

The RPT is so constructed that six samples can be in process of testing at any one time. Therefore one RPT may be considered equivalent to five or six WRT's.

FABRICS——Seven fabrics were used in this comparison. Their structures are presented in Table II. For this work each laboratory was provided with about two square yards of each of the fabrics.

In order to answer one question of the Committee, one of the laboratories ran a comparison of the results of cutting the specimens with dinking dies, scissors and

TABLE VII

RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "D"

Sample	Direction of Test	Recovery Angle WRT (°)	WRT Rank	Recovery Angle RPT (°)	RPT Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	Coeff of Var WRT (%)	Coeff of Var RPT (%)	T-Test of Sig*	Sig Diff†
A	Warp	155	1.5	147	1	— 8	5.46	5.00	3.52	3.40	3.74	Yes
	Fill	155	1.5	145.5	2	— 9.5	3.94	3.68	2.54	2.53	6.13	Yes
B u	Warp	127	6.5	119	8	— 8	10.00	7.96	7.87	6.69	2.18	No
	Fill	127	6.5	124	7	— 3	12.22	4.85	9.62	3.91	0.79	No
B t	Warp	138	4	139	3	1	4.06	8.00	2.94	5.76	0.39	No
	Fill	141	3	133	5	— 8	8.16	4.73	5.79	3.56	2.93	Yes
C u	Warp	91	12	79	11	—12	10.90	10.57	11.99	13.37	2.73	Yes
	Fill	95	11	76	12	—19	6.32	7.91	6.65	10.41	6.51	Yes
C t	Warp	135	5	136	4	1	2.75	8.91	2.04	6.65	0.39	No
	Fill	126	8	126	6	0	13.65	9.08	10.83	7.21	0.00	No
D u	Warp	83	14	43	13	—40	10.62	7.72	12.80	17.97	10.58	Yes
	Fill	88	13	52	14	—36	7.71	4.00	8.76	7.69	14.28	Yes
D t	Warp	118	9	99	9	—19	11.52	16.00	9.76	16.17	3.35	Yes
	Fill	110	10	90.5	10	—19.5	10.02	7.64	9.11	8.44	5.39	Yes

* Critical value for T at the 5% level is 2.306.
† Probability that WRT and RPT crease angles are the same, is high for "No", low for "Yes".

razors. These results presented below in Table III indicate no significant differences between the methods. It is noted, however, that dinking is most rapid and uniform.

OPERATORS——In all but one laboratory there were no operators experienced on the RPT. Some of the laboratories had operators with previous experience in the use of the WRT. There is no evidence, however, that lack of extended experience is a handicap to successful use of either instrument. Laboratory technicians can be taught the use of either instrument in a few minutes.

EXPERIMENTAL RESULTS

In Tables IV through X inclusive are presented the details of the tests performed in the co-operating laboratories.

The differences in the size of the angles measured by each instrument in 71 out of 98 (or 73%) of the cases show the WRT generally measured a larger crease-recovery angle.

The standard deviations as calculated from the actual angular measurements are larger for the WRT in 54 out of 98 cases. This is probably not significant.

The coefficient of variation (standard deviation expressed as a percentage of the mean) for the WRT was larger in 48 out of 98 pairs of measurements. This is probably not significant.

The standard error (the result of dividing the standard deviation by the square root of the number of single tests involved) was found to be larger for the WRT in 34 (63%) out of the 54 pairs of measurements for which complete data were available for this summation. This indicates that any single set of 5 or 6 angular measurements made on the WRT may be slightly less near the true average than a similar set on the RPT.

The probability that both instruments measure the same angle is good in only 37 out of 98 pairs of measurements. This is evidence that the machines do not measure the same angle. This is not remarkable when the differences in time, pressure and other factors are considered.

In all of the tests both machines have demonstrated their ability to differentiate between untreated and treated fabrics by measuring crease-recovery angles.

The instruments have demonstrated their ability to rank fabrics in the same order in the majority of comparisons. In order of decreasing crease resistance the fabrics are A, Bt, Ct, Bu, Dt, Cu and Du.

The RPT Kendall coefficient of concordance was 0.93, while, for the WRT, it was 0.95 for all the laboratories in comparison with the ranking given above. Both instruments appear to be more discriminating in the extremes of angular

TABLE VIII
RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "E"

Sample	Direction of Test	Recovery Angle WRT (°)	WRT—Rank	Recovery Angle RPT (°)	RPT—Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	Coeff of Var WRT (%)	Coeff of Var RPT (%)	Sig Diff†
A	Warp	153.6	1	135.9	3	—17.7	1.56	2.19	1.0	1.6	Yes
	Fill	153.2	2	140.0	2	—13.2	3.81	5.13	2.5	3.7	Yes
B u	Warp	132.7	8	133.0	5	0.3	1.92	3.79	1.4	2.8	No
	Fill	138.5	5	124.4	8	—14.1	4.60	3.78	3.3	3.0	Yes
B t	Warp	145.2	4	142.3	1	— 2.9	3.66	3.82	2.5	2.7	No
	Fill	149.7	3	134.0	6	—15.7	3.23	2.89	2.2	2.2	Yes
C u	Warp	96.4	11	69.8	12	—26.6	5.68	7.39	5.9	10.6	Yes
	Fill	92.5	12	77.2	11	—15.3	5.90	9.84	6.4	12.7	Yes
C t	Warp	137.4	6	134.8	4	— 2.6	4.83	5.70	3.5	4.2	No
	Fill	135.5	7	130.9	7	— 4.6	8.44	7.40	6.2	5.7	No
D u	Warp	68.1	14	48.0	14	—20.1	8.83	10.41	13.0	21.7	Yes
	Fill	82.4	13	51.3	13	—31.1	8.36	12.24	10.1	23.9	Yes
D t	Warp	103.5	10	88.6	10	—14.9	5.52	10.35	5.3	11.7	Yes
	Fill	112.6	9	89.8	9	—22.8	4.68	6.98	4.2	7.8	Yes

* Probability that WRT and RPT crease angles are the same is high for "No", low for "Yes".
Note: All fabric samples were thoroughly conditioned at 65% RH and 70° F prior to testing. Sample size was 1 x 4 cm. Twelve tests in each direction made on each fabric on each machine.

TABLE IX
RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "F"

Sample	D*	Recovery Angle WRT (°)	WRT—Rank	Recovery Angle RPT (°)	RPT—Rank	RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	% Coeff of Var WRT	% Coeff of Var RPT	Std Error WRT (°)	Std Error RPT (°)	T-Test of Sig**	Sig Diff†
A	W	148	2	152	4	4	2.0	4.1	1.35	2.73			4.25	Yes
	F	156	1	154	1	— 2	1.8	3.6	1.13	2.35			2.43	Yes
B u	W	127.5	8	136.7	8	9.2	4.8	4.9	3.7	3.6	1.37	1.42	6.18	Yes
	F	136.8	7	139.9	7	3.1	3.5	2.7	2.0	1.95	1.00	0.79	3.31	Yes
B t	W	137.9	6	153.4	2	15.5	2.4	1.7	1.7	1.10	0.69	0.49	24.8	Yes
	F	142.3	4	153.1	3	10.8	3.9	3.2	1.8	2.1	1.12	0.52	10.1	Yes
C u	W	89.8	12	92	12	2.2	8.9	6.4	9.9	6.9	2.56	1.84	0.94	No
	F	101.3	11	97.9	11	— 3.4	5.6	2.6	5.5	2.65	1.62	0.75	3.28	Yes
C t	W	140.3	5	148.2	6	7.9	6.4	4.9	4.6	3.32	1.85	1.42	4.65	Yes
	F	142.9	3	151.5	5	8.6	3.6	3.5	2.5	2.29	1.02	1.01	8.17	Yes
D u‡	W	79.6	14	59.5	14	—20.1	5.1	11.0	6.43	18.52	1.47	3.18	7.46	Yes
	F	88.3	13	73.1	13	—15.2	5.3	3.8	6.02	5.14	1.53	1.08	9.7	Yes
D t‡	W	118.0	9	102.2	10	—15.8	6.1	3.5	5.15	3.41	1.75	1.01	11.95	Yes
	F	117.0	10	106.5	9	—10.5	6.3	3.0	5.42	2.85	1.83	0.87	8.01	Yes

** Critical value for T at the 5% level is 2.07.
† Probability that WRT and RPT crease angles are the same, is high for "No", low for "Yes".
* D equals direction of test, warp and filling.
‡ Identification tickets on checked fabric appear to have been switched when received. In the above table they have been, it is believed, correctly identified.

TABLE X

RESULTS OF COMPARISON OF MONSANTO WRT AND CYANAMID RPT IN LABORATORY "G"

Sample	D*	Recovery Angle WRT—Rank (°)		RPT—Rank (°)		RPT minus WRT	Std Dev WRT (°)	Std Dev RPT (°)	% Coeff of Var WRT	% Coeff of Var RPT	Std Error WRT (°)	Std Error RPT (°)	T. Test of Sig†	Sig Diff‡
A	W	157	2	150	3	— 7	6.6	3.3	4.20	2.20	1.91	0.95	4.58	Yes
	F	160	1	152	2	— 8	4.5	3.8	2.81	2.50	1.30	1.10	4.17	Yes
B u	W	149	7	130.4	8	—18.6	2.6	3.4	1.74	2.61	0.75	0.98	13.84	Yes
	F	153.5	5	143.4	5	—10.1	3.4	2.9	2.21	2.02	0.98	0.83	7.52	Yes
B t	W	156	3	145.6	4	—10.4	2.6	3.7	1.67	2.54	0.75	1.07	10.75	Yes
	F	155	4	154.4	1	— 0.6	2.9	3.4	1.87	2.20	0.83	0.98	1.53	No
C u	W	108	14	84.3	11	—23.7	6.3	6.9	5.83	8.11	1.82	1.99	8.23	Yes
	F	117	11	82	12	—35	6.7	6.4	5.72	7.80	1.94	1.85	12.52	Yes
C t	W	148	8	140	6	— 8	4.6	7.7	3.11	5.50	1.33	2.23	3.21	Yes
	F	151	6	138.5	7	—12.5	7.4	6.5	4.90	4.69	2.14	1.88	4.21	Yes
D u	W	112	13	48.4	14	—63.6	4.7	5.3	4.19	10.95	1.36	1.53	30.37	Yes
	F	112	12	54.2	13	—57.8	4.7	5.7	4.19	10.52	1.36	1.65	26.07	Yes
D t	W	130.5	10	93.2	10	—37.3	11.9	6.3	9.12	6.75	3.44	1.82	9.19	Yes
	F	137	9	98.8	9	—38.2	5.4	4.9	3.94	4.95	1.56	1.42	17.69	Yes

* D equals direction of test, warp and filling.
† Critical value for T at 5% level is 2.07.
‡ Probability that WRT and RPT crease angles are the same is high for "No", low for "Yes".

values.

Respectfully submitted,
COMMITTEE ON EVALUATION
OF DURABLE FINISHES
MARTIN H GURLEY, JR
Chairman

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ACKNOWLEDGMENTS

American Cyanamid Company
American Textile Company, Inc
Bates Manufacturing Company
Esselen Research Corporation
Fabric Research Laboratories, Inc
Monsanto Chemical Company
J C Penney Company, Inc
Philadelphia Quartermaster Depot
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Piedmont Section——

NEW DEVELOPMENTS IN DYEING CELLULOSE-ACETATE YARN*

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Tennessee Eastman Company, Kingsport, Tenn

D G Carmichael

After an historical introduction, the authors point out that cellulose acetate is now dyed mostly with water-insoluble but highly dispersible dyes, which are either azo combinations or aminoanthraquinone derivatives. Many of these dyes, especially the fast-to-light blues, are subject to atmospheric gas fading, but can be considerably protected with special antifume agents. Some newer blue dyes are now available that are not subject to gas fading and that are adequately light-fast for draperies.

Most of the paper explains how to apply naphthols and fast color bases simultaneously on acetate yarn in circulating machines then to diazotize the base and permit coupling in a second step. This special technique differs from the method for rayon and cotton. Such dyeings display very good fastness to both light and washing.

W R Ivey, Jr

INTRODUCTION

ALTHOUGH cellulose acetate was prepared in 1865 by Schuetzenberger *(1)* who heated cellulose in a closed tube with acetic anhydride, cellluose acetate for forty years remained only within the sphere of academic interest. In the interim, important advances and studies were made by such investigators as Cross and Bevan, G W Miles, A D Little, W H Walker, H S Mork and H and C Dreyfus. Two of the retarding factors in the commercial development of this product were poor tensile strength and brittleness of films made from a triacetate, which was soluble in chloroform but insoluble in acetone. An important technical advancement was made by G W Miles *(2)*, who found that, by partial hydrolysis, a cellulose acetate was soluble in comparatively cheap solvents, such as acetone.

During World War I, large quantities of cellluose acetate were used in the preparation of airplane dopes in England; and, at the close of the war, attention was given to using this material for the production of a fiber. Large-scale production of cellulose-acetate fiber was started about 1920-21, one of the pioneers of which was the Lustron Company of Boston.

* Presented by William R Ivey, Jr, before the Piedmont Section at the Robert E Lee Hotel in Winston-Salem, N C, on Feb 2, 1952.

EARLY DYEING OF ACETATE

The most difficult problem encountered in the early days by the cellulose-acetate-fiber industry was dyeing, which retarded commercial development somewhat. The first attempts to dye cellulose acetate were made with the known classes of dyestuffs from the acid, mordant and basic groups. It was noted that dyestuffs of a basic character and those containing hydroxyl groups had a perceptible affinity, while the presence of sulphonic acid groups reduced affinity. With the limited number of dyestuffs and the incompatibility of the different classes with each other, combination shades were most difficult, and the fastness to light, crocking and washing was of a low rating. Methods were developed to employ acid and direct dyestuffs in solvents or swelling agents, but the cost of dyeing made these processes impractical. One of the earlier steps taken in overcoming the difficulty of dyeing was saponification of the cellulose-acetate fiber with alkalies and subsequently dyeing with cotton and viscose dyestuffs. More even dyeing results were obtained by replacing the irregular and energetic action of caustic soda with milder alkalies and by addition of neutral salts to reduce their action.

SPECIAL DYES

IONAMINES——The first development in special dyestuffs for cellulose acetate fibers was the Ionamines discovered by Green and Sanders *(3)*. The Ionamines are water-soluble omega-sulphonic acids of aminoazo and aminoanthraquinone compounds. The Ionamines, during dyeing, are gradually hydrolyzed by an addition of acid or alkali, and the regenerated insoluble azo compounds are absorbed by the cellulose-acetate fibers. Ionamines produced from a primary amine could be diazotized on the fiber and converted to shades of orange, scarlet, red, maroon or black by the use of β-naphthol, β-hydroxynaphthoic acid, resorcinol or other couplers. Difficulty was encountered with different rates of hydrolyzation in mixtures, which caused trouble in matching shades, and the dyeing time was too lengthy.

The most important and widely used dyestuffs at present are the dispersed acetate dyestuffs, which are highly dispersed azo and anthraquinone compounds. It has been known for more than a decade that blue acetate dyestuffs are likely to fade while on display or in storage. This fading is due to the effect of oxides of nitrogen in the air, produced by combustion of coal, gas, or other fuels, by the use of electrical heating elements or other red

hot materials in the air. Antifume agents have been developed and used successfully to inhibit this type of fading, but these finishes are not the solution to the problem.

Eastone Blue BGF was the first blue acetate dyestuff to withstand gas fading successfully but lacked the high light fastness necessary for drapery, upholstery and men's wear fabrics. Eastman Fast Blue GLF Conc is the first atmospheric gas-fast blue with excellent fastness to light and also very good fastness to perspiration, sublimation, hot pressing, urea-formaldehyde resins and dry cleaning. There is also a bright greenish yellow, violet, red and reddish yellow in this gas and light fast GLF series.

NAPHTHOLS ON ACETATE

It has been desirable for some purposes to dye cellulose acetate with colors possessing wash fastness greater than that provided by acetate dyestuffs. There has long been the opinion that naphthol colors might show the greatest promise if a satisfactory and practical procedure could be established for dyeing. The application of these insoluble azoic or naphthol colors to cellulose acetate is not new and is mentioned in literature by J Pokorny *(4)*, who, prior to 1923, solubilized para-nitroaniline and beta-naphthol together and dyed cellulose acetate in the same bath. Coupling of the components took place after diazotization of the para-nitroaniline with nitrous acid. R Metzger and H Rohling *(5)* have described methods of dyeing naphthols and bases together and then completing the coupling by diazotization. Difficulty has been experienced in the application of naphthols to cellulose acetate in package form because of the filtration of the bases. A great amount of work has been done along these lines recently, and, as a result, a method of naphthol application on acetate package yarn has been developed and has given satisfactory penetration and levelness without filtration on a practical scale.

At the outset of this work, a number of factors had to be explored; the variables, existing in each one, narrowed to those giving the very best results. The first factor, naturally, was in the selection of the naphthols themselves. It was necessary to find the ones with the best affinity for acetate fiber. These naphthols should possess the greatest solubility so that a minimum amount of alkali could be used in the dyeing; this would be important to minimize any danger of saponification. Another factor in the selection of naphthols was in using those that would provide a good workable range of shades with various bases to give the best possible fastness properties. Investigation proved that Naphthol AS-D provided the answer to most of these problems with Naphthols AS-BG, AS-LB, AS-G, and AS-OL filling gaps existing in the range of shades.

The method of dyeing the naphthol was limited. The standard cold-dissolving method was used to prepare the naphthol solution, which is as follows:

1 part of naphthol is pasted with
2 parts of denatured alcohol and
¼ part of caustic soda dissolved in
2½ parts of water added very slowly with stirring until in solution. This naphthol in solution has an affinity for acetate, but, by the addition of ½ to 1% of monosodium phosphate, a fine dispersion is obtained, which has an even greater affinity.

The next matter to be considered was the preparation and dyeing of the amine, or fast color base. The problem of dispersing the base so that it would not tar or filter out on package yarn was finally solved by the use of a nonionic dispersing agent. The best method was found to be as follows:

1 part base is pasted with
2 to 3 parts denatured alcohol and
2 parts nonionic dispersing agent is added. This mixture is stirred into 25 parts cold water. In the case of Blue 2B Base, hot water at 160° F is used in place of cold water.

In experimental work on dyeing temperatures, it was learned that 160° F provided the best build-up and exhaustion of the naphthols and fast color bases on the acetate fiber. At this established temperature, a dyeing period of 30 minutes gave maximum results.

The best fast color bases were found to be

Scarlet R Base	Garnet GBC Base
Red KB Base	Blue 2B Base
Red PA Base	Eastman Diazo Black RK
Red B Base	m-aminobenzotrifluoride

The diazotization or coupling of the naphthol and the base within the acetate fiber is carried out with conc hydrochloric acid in amounts ranging from 6 g/liter (¾ oz/gal) for yellows, oranges, browns, scarlets, reds, bordeaux, and blacks to ¾ g/liter (1/10 oz/gal) for blues and with sodium nitrite in amounts ranging from 3 g/liter (⅜ oz/gal) for yellows, oranges, browns, scarlets, reds, bordeaux, and blacks to 2 g/liter (¼ oz/gal) for blues. A 10-minute diazotization at 160° F gives optimum results and longer periods show no improvement in shade or ultimate fastness.

The final development of these naphthol colors cannot be emphasized too greatly. It has been found by a great amount of experimentation that the after soaping of these dyestuffs on cellulose acetate will determine the fullness and bloominess of the shade and the degree of light and wash fastness that the dyeings will possess. A thorough soaping at the boil for 15 minutes with 3 g/liter (½ oz/gal) of soap, ½ g/liter (1/16 oz/gal) of soda ash and 1 g/liter of pine oil (⅛ oz/gal) is prescribed for all shades except blues. For blues, 4 g/liter (½ oz/gal) of a nonionic and 1 g/liter (⅛ oz/gal) of pine oil should be used.

MILL PROCEDURE——A typical mill procedure for the dyeing of naphthol colors on packaged acetate yarn, which will vary slightly from the methods used on other fibers, would proceed as follows: The machine is filled to volume with water, and 1% of a synthetic detergent is added. The prepared naphthol solution is strained into the machine and circulated well. The packaged yarn is entered dry, the machine closed, and circulation is started inside-out, the circulation cycle running 10 minutes inside-out and 5 minutes outside-in. The temperature is raised to 120° F, the dyeing is run for 10 minutes, and then the strained prepared color base is added. The temperature is raised to 160° F, and ½% monosodium phosphate for filament yarn or 1% for spun yarn is added. After the addition of the phosphate, the dyeing is run for 30 minutes. At the end of this time, the bath is dropped and the yarn is rinsed for 10 minutes in cold water containing 20% of common salt, owf, and then in cold running water for 5 minutes.

The machine is again refilled, and circulation is started inside-out. Heat to 120° F and add the diluted hydrochloric acid and dissolved sodium nitrite. Raise the temperature to 160° and diazotize for 15 minutes. Drop the bath and give a cold running rinse for 5 minutes.

Refill the machine and heat to 120° F; add soap, soda ash and pine oil, or nonionic and pine oil in the case of blues, and raise the temperature to a boil. Soap for 15 minutes at this elevated temperature; drop the bath and rinse for 10 minutes at 160° F and then with cold running water for 5 minutes. The packages are then removed, extracted and dried.

From exhaustive tests run on these dyeings, we find that a resistance of 40 to 100 hours of exposure in a Fade-Ometer can be obtained, depending on the particular combination of naphthol and base. Very good (4) resistance to washing is also achieved with such dyeings, that is, no loss of strength or staining from the No 3 AATCC test of 45 minutes at 180° F with a concentration of 0.5% of soap and 0.2% of soda ash.

In looking at this dyeing method from

(Concluded on Page P428)

South Central Section——

NEW DYEING METHODS*

C C CAYCE
General Dyestuff Corp

In a talk for which it was announced that new dyeing methods would be discussed, the speaker could hardly avoid devoting most time to the new machines in which the new methods of dyeing are carried out. Molten-metal ranges and Williams units were only briefly mentioned, but the hot-oil dyeing method in the improved Williams unit was explained at some length, and especially its usefulness for the application of vat and sulfur dyes. The speaker then pointed out how the necessity of temperatures above 220° F for dyeing some of the new wholly synthetic fibers has forced the improvement of package machines so that they can be operated at pressures well above atmospheric. Enclosed jigs are also fact becoming common because of the need for higher and more uniform temperatures throughout a roll in jig dyeing. The new fiber-dyeing machine invented by S H Williams and based on the original Williams unit for piece goods was next given briefly, and the advantages of dyeing fibers continuously were pointed out. Methods for dyeing Dacron polyester fiber and Orlon acrylic fiber were explained, and lastly, the use of the Marhen Process for the control of vat dyeing.

OUR subject, "New Dyeing Methods", covers a very broad span and, since the textile industry has no common exchange to disperse all the new methods that you ingenious technicians arrive at during your daily duties, we can only touch on some of the practices that are known to us.

In this approach, we have divided the discussion into two categories, namely:

(1) New Machines for Dyeing, and

(2) New Methods for Dyeing.

NEW CONTINUOUS PIECE-DYEING EQUIPMENT

All of you are familiar with the pad-steam process *(1)* and the utilization of Williams units *(2)* for continuous dyeing, so I will not subject you to a dissertation on these subjects. The two recent new methods for the continuous application of color to piece goods is the Molten-Metal Process *(3)*, originated in England, and the Hot-Oil Process *(4)* in the new high-pressure Williams units.

I am not competent to talk on the Molten-Metal Process as I have not seen the machine in action, and I shall not merely repeat what has been published, as that has been done often enough already.

I do feel competent, however, to discuss the Hot-Oil Process since the General Dyestuff Corporation is the inventor. This method for dyestuff application has, by now, been accepted by a number of plants as being the best procedure to process difficult shades and fabrics which, heretofore, have not lent themselves to continuous dyeing application and had to be jig-dyed.

It is not my intention to imply that the Hot-Oil Process is the ultimate or should be used on all continuouly dyed shades. There are a number of colors that process equally well in a water medium and for which oil shows no advantage. Since the oil industry is not an eleemosynary institution, the cost of the petroleum is a necessary factor. The main advantage of the hot-oil method is that of giving the dyer controlled continuous operation for depths of shade that formerly fell in the pad-jig category. We are referring to heavy shades of vat greens, navies, browns and royal blues that do not produce money value or good shading when dyed in a water bath.

The Hot Oil Process is new to continuous dyeing only in the development bath, that is, the goods are padded with vat pigment, dried and then treated with caustic soda and hydrosulfite at temperatures ranging between 100 and 120°F in accordance with the nature of the fabric. From the chemical bath, the goods enter the hot-oil chamber. The temperature in this bath ranges from 220 to 235°F. A complete flash development takes place, and then the goods enter the next box containing a suitable emulsifying agent to remove the oil from the cloth. The next steps of oxidation, soaping, etc, follow conventional lines.

The Hot-Oil Method has the following advantages for the application of vat dyes:

1) Low-temperature chemical bath, which enhances control as no breakdown of color occurs.

2) Dyestuff consumption cut 10-20%.

3) Hydro consumption cut 25-50%.

4) Shade arrived at more quickly, as equilibrium of color in chemical bath is established sooner because of less stripping at lower temperatures.

Sulfur colors, normally not such good actors when dyed continuously, take to the Hot-Oil Method like Samson to Delilah. The dyestuff is padded at 180 to 200°F, and the padded goods enter the oil unit wet. Unbelievable savings have been effected with dyestuff formulations cut from 40 to 60% over regular continuous methods. Bronzing and cross-shading have also been eliminated. The new high-steam-pressure (50-pound) Williams unit is best suited for the Hot-Oil Method because it permits the high temperature needed for successful operation.

NEW MACHINERY FOR PACKAGE DYEING——With the advent and resultant headaches of the new synthetic fibers, such as the polyester fiber, the acrylic fiber, the polyamide, and others, new mechanical ideas had to be forthcoming. Since a number of these fibers are resistant to dyestuff at temperatures near or at the boil, pressure dyeing has become necessary *(9)*. A conventional package machine can be equipped to provide temperatures of 240-260°F. This is the only radical change required in the design of package-dyeing equipment.

In connection with these new synthetics on woven piece goods, enclosed jigs, where higher temperatures can be maintained, are fast becoming popular dyehouse equipment.

CONTINUOUS FIBER-DYEING MACHINE——A machine to dye fibers continuously has long been past due for the textile industry. General Dyestuff Corporation has again come up with equipment that has a brilliant future *(5)*. This machine is designed to handle stock, laps, sliver, tow, roving, warps and nonwoven fabrics without tension.

The principle of dyeing or applying treating solutions to the above mentioned

* Presented at the Hotel Patten in Chattanooga, Tenn, before the South Central Section on Dec 21, 1951.

materials is the same as that of the Williams units for piece goods, namely, of utilizing small volumes, only one well and one removable baffle being contained in the continuous fiber machines, as compared with four wells and four removable baffles in the piece-goods unit. The volume of the unit of the fiber machine depends on the width or face of the machine. For example, in a 40-inch-width machine, the volume of liquor contained in the well is 50 gallons.

The fiber to be treated is conveyed through the machine by endless porous belts, or conveyers, throughout the entire processing without tension on the fiber. These belts are so constructed of stainless steel as to allow free movement of the treating solutions and to insure maximum life of the belts themselves. The loose fibers are fed to the two coacting belts from a feeder. The fibers are carried by the bottom porous belt up to the entering point of the machine. This bottom conveyer belt is met by another endless co-acting belt, and the fiber is carried between these two belts down through a well containing the treating liquor and is thoroughly saturated. The belts go under a guide roll, or submersion roll, attached to the bottom of a removable liquor-displacing baffle and back up the opposite side of the well. The two belts carry the fiber between two light squeeze rolls, which return most of the excess liquor to the well. At this point, the belts separate, feeding the fiber on to another endless belt, which, in turn, carries it into two heavy squeeze rolls. Here, the stock is extracted to the desired pickup of the treating liquor. The excess liquor returns to the well. The stock is fed by these rolls on to another endless belt, where it is washed or chemically treated as desired. It is again squeezed and fed on to another endless belt for subsequent washing. The fiber finally passes through heavy nip rolls, which act as extraction rolls, and the stock then falls into a hopper in front of the dryer.

The foregoing is a brief synopsis of the so-called single-well machine, which was the first machine manufactured and, at present, is located in General Dyestuff's office building in Charlotte, N C. After experimenting for several years, we found a number of stocks could be processed with just a single-well machine, but, to make the machine more versatile, a two-well machine was built by Morrison and has been delivered to a textile plant located here in the South. The construction of the two-well machine has the following continuity:

A first dye well and an apron carrying the stock to the first nip roll, and two more sections for various aftertreatments. From the third nip roll, the stock then enters another dye well and three identical aftersections for aftertreatment, etc.

By utilizing two solution-treating wells with nips in between, we are able to dye naphthols continuously, where, otherwise, it would have to be a two-run proposition with only one dye well. Also, for scouring woolens or other oily fibers, the two-well treatment is more effective in a scouring and cleansing operation.

A typical continuous-dyeing operation in this machine would be as follows:

In the case of dyeing vat colors on a given fiber, the dye solution is put into the first well. The stock is carried through this reduced vat color by the porous belts, and then it is washed by the use of spray pipes while the fibers are traveling through the next three sections. The stock then enters the second well, in which any preferred oxidizing agents are applied. Then the stock is cold-washed in the first spray section, after the second treating well, and then hot-washed in the remaining two sections with heavy spray.

In the case of dyeing naphthol colors, a naphthol is applied in the first dye-well, extracted through the next three sections, and a Fast Color Salt or diazotized base is applied in the second dyeing well, the stock then being thoroughly washed in the remaining three sections before going into the delivery nip of the machine.

Sulfur dyestuffs are applied by putting dissolved sulfur dye in the first well and chrome and acid in the second well. The oxidized dyeing is rinsed in the sprays.

Direct dyestuffs can be applied by putting dissolved direct color in the first well and boiling salt in the second well.

Some of the operations that have been successfully carried out, on a small scale, on this new continuous range are the following:

1) Dyeing of light, medium and full shades of Helindon colors on wool, stock, laps and tow.

2) Dyeing of bright red shades of naphthols on cotton, rayon, or wool stock, as well as on rayon tow.

3) The dyeing of vats on cotton, rayon, nylon, and other types of stock, as well as on cotton sliver, or rayon and Vicara tow.

4) The scouring of wool in the grease and the scouring of oily cotton waste.

5) The dyeing of cotton, rayon, and nonwoven fabrics with direct colors.

6) Treating cotton wadding with permanent flame-proofing materials.

This list of operations is by no means complete, and some of those listed have been done on only a few pounds to determine the practicability of using the machine for the purposes mentioned. At this time, we do not have any practical or large-scale production figures since our lack of facilities in Charlotte makes it impractical to try to process any large poundage.

The advantages of this new method of fiber processing are obvious. The speed of the machine varies from 4 to 30 yards per minute. A two-pound-per-yard lap can be successfully handled. At the slowest speed, 250 pounds of stock can be processed per hour. If the top speed were maintained, 3,000 pounds per hour could be run through the machine, but the average will probably be about 10 yards per minute, or 1,200 pounds per hour. This figure, however, depends upon the width of the machine and the density of the web carried through by the belts. The speed of the machine will also necessarily depend on the drying and other equipment for handling the fiber after it has been processed, as well as on the type of fiber and the process.

With increased production, the labor cost should drop sharply. In conventional batch dyeing, the dropping of a relatively high percentage of treating chemcials and dyestuff down the drain increases costs considerably. In this continuous process, the loss of chemicals and dyestuff is cut to a minimum. Only the liquor remaining in the well after the run is completed is lost. On long runs, this loss should be insignificant.

The machine is very compact in design, and a space of only about 39 feet is necessary for a two-well machine. The width of the machine depends upon specifications and would be 50, 60 or 70 inches. Very little steam is consumed in operating this machine as only a small volume of treating solution is heated, and the wells are steam-jacketed and well insulated.

This brief outline should give you some insight as to what the future holds for the processing of loose or nonwoven fibers.

NEW METHODS OF DYEING

I can hardly do justice to new methods of dyeing in the time available, even if I knew them all. So many formulations and techniques are appearing weekly that what you think is new today actually is just a stop-gap until a better method is turned out of the minds of the technicians.

New synthetics have brought about the most radical changes in dyeing methods *(8)*. For example, Dacron polyester fiber can be dyed with certain acetate colors in light or medium shades by boiling for an hour. A fuller range and deeper shades can be dyed at 250° F under pressure. Since a number of fabrics cannot be dyed under pressure, a method had to come forth to handle Dacron piece goods. This was accomplished by the use of swelling agents. Two procedures that we are familiar with are the paraphenylphenol method and the monochlorobenzene method. The quantity of the paraphenylphenol necessary

varies with the depth of shade, running from 1 to 6% and, in some cases, up to 9%. Paraphenylphenol is dissolved in one-third as much caustic soda and added to the dyebath. The goods are treated in this bath for 5 to 10 minutes at room temperature. The paraphenylphenol is then thrown into a fine precipitation with 4% acetic acid, 56%. The color is added, and then the bath is raised to the boil and boiled for 1½ hours. Dyeing is followed by rinsing.

The monochlorobenzene method is only applicable to closed machines as the monochlorobenzene is very volatile. Twenty percent of monochlorobenzene on weight of fiber is made into a water emulsion with one-tenth its weight of Monopol Brilliant Oil 48%. The goods are treated for 15 minutes at room temperature. The monochlorobenzene emulsion will exhaust on the fiber at this temperature. The color is added and the temperature is raised to 190° F. Color passes from the water phase into the monochlorobenzene, for which it has affinity. It is transferred from there onto the fiber. The temperature of 190° F is held for one hour. To dispel the monochlorobenzene from the fiber in the bath, the temperature is raised to 205° F and held for one hour.

There are two main drawbacks to this method:

1) The monochlorobenzene is toxic and must be handled very carefully, and good ventilation must be maintained.

2) This chemical is so volatile that it cannot be used on piece goods where the dyeing is exposed, otherwise it will evaporate rapidly.

ORLON ACRYLIC FIBER——ORLON, likewise, has its dyeing problems *(7)*. A suggested formula for type-41 Orlon acrylic fiber is as follows:

Add 10% copper sulfate to the dyebath, which contains the thoroughly clean material. This addition should run for about 5 minutes at 140° F. Thereafter, add 4% hydroxylammonium sulfate and run for 5 minutes longer at the same temperature. Then add well-dissolved dyestuff, raise the temperature slowly to the boil and boil for one to 1½ hours. If additions are necessary, cool the bath down to 180° F, make a color addition, raise the temperature again to the boil and boil for 1½ hours longer.

MARHEN PROCESS —— One of the newest methods for the control of vat dyeing is the Marhen Process, sponsored by the General Dyestuff Corporation *(6)*. This process gives the dyer a simple method for the electronic measurement of the reducing potential of a vat dyebath, and also shows how this potential can be controlled. As a result, both excesses and deficiencies may be avoided.

In the reduction of a vat dye, caustic soda and hydrosulfite develop a potential source of electrons. By use of suitable electrodes and measuring equipment, the potential ortendency to liberate electrons resulting from the reaction may be measured. This measure is then the reduction potential of the dyebath, or the potential availability of electrons for reduction of a vat dye.

A knowledge of the hydro-caustic relation in a dyebath eliminates guess work and is much more positive and faster than titration methods. This latter procedure only reflects excess hydro and caustic in the bath which, of course, is not the important factor and is also wasteful.

The instruments required for the measurement of reduction potential are simple to operate and not too expensive. A set of electrodes, comprised of one platinum and one calomel, are immersed in the bath to be measured. These electrodes are connected in turn through a high-impedance amplifier to a recording potentiometer. Reduction potential is recorded directly on this potentiometer.

We have successfully demonstrated this machine in a number of plants in the country, and, in every instance, the operation has been very successful. Better shading from end to end on cotton piece goods and a saving in hydro are the most outstanding advantages. In cases where chemical balances were set up wrong, better color value was received when the correct potential was established by proper adjustment of hydrosulfite and caustic-soda concentrations. We see a big use for these instruments in all forms of vat dyeing, such as continuous piece-goods machines, jigs, becks, package machines, rawstock-dyeing and skein-dyeing equipment.

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New Developments—

(Concluded from Page P425)

a cost point of view, it will compare very favorably with the same shades run on other fibers. Dyeing time, materials handling, amounts of dyes and chemicals are approximately the same.

Successful and practical naphthol combinations for a range of shades on acetate with good fastness to light and washing include the following:

Yellows
Naphthol AS-G
Fast Red B Base
Naphthol AS-G
Fast Red KB Base

Oranges
Naphthol AS-D
m-aminobenzotrifluoride
Naphthol AS-BG
Fast Orange R Base

Scarlet
Naphthol AS-D
Fast Scarlet R Base

Reds
Naphthol AS-D
Fast Red B Base
Naphthol AS-D
Fast Red PA Base

Bordeaux
Naphthol AS-D
Fast Garnet GBC Base

Browns
Naphthol AS-LB
Fast Red B Base
Naphthol AS-LB
Fast Red PA Base
Naphthol AS-LB
Fast Garnet GBC Base

Blue
Naphthol AS-D
Fast Blue 2B Base

Navy
Naphthol AS-BG
Eastman Diazo Black RK

Black
Naphthol AS-D
Eastman Diazo Black RK

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Hudson-Mohawk Section—

NONWOVEN FABRICS*

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H E Shearer

Nonwoven fabrics, which cost much less than woven materials, or only 4 to 30c per yard, are manufactured from webs of interlocking fibers. Webs are prepared from various fibers and fiber mixtures, which are held together by bonding materials. Bonding may be accomplished with admixed thermoplastic fibers like acetate or Vinyon, which become plastic and thus adhesive by application of heat; or by treatment with solutions of solvent- or water-soluble substances that leave a pliable adhesive film upon drying. Mechanical features are also involved in bonding. The use of solvent on solvent-soluble fibers like acetate is claimed to be least practical. Besides explaining bonding, the author discusses especially the formation of webs. The future possibilities of nonwoven fabrics receive the author's concluding attention.

INTRODUCTION

VARIOUS news releases emanating from more or less reliable sources over the past few years have stimulated an unprecedented interest in a material that has come to be known as both "nonwoven" and "bonded-fiber" fabric. This material is not woven, knit, or spun, but is built up by the interlocking of fibers in the form of a web or mat, and the bonding in place of these fibers by mechanical work, chemical action, moisture or heat. It has been defined by a recently established Sub-Committee B-19 of ASTM Committee D-13 as "a structure consisting of a web or mat of fibers held together with a bonding material".

HISTORY

Nonwoven fabrics had their beginning many years ago in the more or less heavy and nonappealing bonded-cotton waddings and wool felts, which bear little resemblance to the fabric of today. From these earliest materials were first conceived the lightweight, hard-pressed, bonded webs of paper-like texture which were looked upon with disdain by textile weaver and paper manufacturer alike. However, as a new fabricated material with characteristics built in that are common to neither woven fabric nor paper, these bonded webs are now being utilized in the development of original products in their own right. They have also developed into soft, absorbent, lofty and drapable materials that may be processed to fit into many of the established fine-specialty-paper and woven-fabric uses. It is the emergence of this material with improved fabric-like qualities that has recently stimulated the interest of both manufacturers and consumers, and this material is now making rapid strides as a textile in many household and industrial applications *(1)*.

The production of these nonwoven fabrics has increased from a few thousand pounds in 1945 to an estimated present production of around 15,000,000 lb with the prospect of this figure being steadily increased in the years to come, based on the sights set by the present producers in this field. There are now about ten manufacturers very active in this field, with as many more conducting experimental or pilot-plant operations with the thought of becoming commercial producers. Among the latter may be included many long-established manufacturers of heavy waddings, batting or filling materials, who are modifying or increasing their line of products to fit into this modern conception of nonwoven materials.

The introduction of rayon into nonwoven fabrics, the development of improved adhesives, and the refinement of manufacturing techniques have accounted for much of the advance and increased acceptance noted. Major improvements that still face the industry are to increase transverse strength and to reduce the papery appearance and hand. Definite advancement in these directions is being made.

MANUFACTURE

WEB FORMING EQUIPMENT——In the manufacture of nonwoven fabrics the first major and distinctive operation is the formation of the fiber web, which constitutes the basic structure of the fabric. This is usually accomplished on cotton or woolen cards, garnetts, or on special adaptations of these employing aerodynamic principles.

The production of carded fiber from a cotton card for yarn-spinning purposes usually ranges around 10 to 20 lb per hour. It is not unusual for manufacturers of nonwoven fabrics to increase this rate to 35 or 50 lb an hour and to use from 3 to 15 cards in series or in parallel to produce the weight of web desired. Weights of nonwoven fabrics, as produced today, usually range from ½ to 8 oz per square yard with the major production around 1½ to 2 oz.

The width of nonwoven fabrics is usually limited by the width of the web-forming machine. In this respect, woolen cards and garnetts have an advantage over cotton cards. Up to 78" widths may be produced by such means, or, if it is preferred, the web may be slit and doubled to narrower widths of heavier weight with a saving in the amount of equipment required. The higher production rates of woolen cards and garnetts may also be something of an advantage, as well as the wider range of fiber diameters, lengths, and weights of web that they can handle. Attachments are also available for these machines for taking off the web in a cross-laid form. This reduces the number of machines required for producing heavyweight materials and more equally distributes the strength in the finished product. A garnett and cross-feed arrangement are thus operated together in the production of either lightweight or heavyweight fabrics.

Cross-laying the web provides a means of overcoming what has been one of the chief objections to nonwoven fabric, namely, low transverse strength. However, present methods of cross laying

* Presented before the Hudson-Mohawk Section in Albany, N Y, on December 7, 1951.

usually create uneven bands or streaks in the fabric, which, in lightweight material especially, may prove objectionable.

A lack of transverse strength in straight carded webs and unevenness in those produced by conventional cross-feeding methods were early recognized as major retardents to the expansion of nonwoven fabrics into many fields of use. These deficiencies have led to many efforts and to several successful developments of machinery, for overcoming these conditions. The Draper cross-laid web machine was designed with this in mind. It utilizes card lap and makes a product resembling woven fabric in appearance and strength more closely than do many other machines.

The preliminary processing cost of producing card lap, as well as the prospective cost of the machine, has given rise to some skepticism as to how extensively this method may be adopted when the machine becomes generally available. We understand it is now undergoing extensive production trials in the plants of several established manufacturers of nonwoven fabrics.

Our Textile Research Department in American Viscose Corp has developed what is believed to be a rather novel and practical means of producing a cross-laid straticulate nonwoven material utilizing a modification of a single-hopper feed, two-cylinder garnett and the conventional cross-laying and compression-roll attachment. By this method one or more dividers are placed in the hopper feeder so as to form bins into which fibers possessing different desirable characteristics may be placed. It is then possible to feed these in direct rows through the garnett in such fashion that, during the cross-laying of the web, the particular fiber desired may appear solely on the interior of the mat or on either or both surfaces. For instance, a mat may be made with 2/3 of its thickness of rayon or of a rayon-Vinyon blend and the other 1/3 entirely of Vinyon. By locating one or more Calrod heating units on the compression-roll attachment, sufficient heat may be directed at the thermoplastic fiber in the mat to give the density and degree of bonding that is desirable for many nonwoven fabric applications. Heretofore it has been customary to perform such an operation through use of a multiple-card arrangement.

Several methods have been developed and are in commercial use in this country employing at least partial carding and aerodynamic principles for producing randomly dispersed fiber webs having uniform strength in all directions. For those who do not wish to build or develop their own equipment for producing a randomly dispersed web, there is on the market a machine developed especially for the production of this type of material from loose fiber stock. For use in conjunction with the web-forming portion of the machine, there has also been developed an automatic hopper feeder. These both employ aerodynamic principles of operation in a composite unit, which is now known as the Rando Feeder and Webber.

The automatic fiber-feeder portion of this machine is available as a separate unit, and is so designed that it may operate not only with the random-web machine but with other carding equipment as well, which ordinarily uses a hopper feeder or picker for textile purposes. The composite random-fiber-web machine was designed primarily to handle waste fibers of all types, though it may equally well be adapted to handling first-quality rayon staple. It produces webs or mats ranging from ½ to 10 oz per sq yd at a rate of 10 to 40 lb per hour, depending upon the fiber and weight of web produced.

TYPES OF FIBER USED——Rayon, acetate, Vinyon, cotton, wool, and blends of these are commercially used in the manufacture of nonwoven fabrics. Other fibers may be used, though not all will be found to process equally well on each type of equipment. The particular fiber used on any one machine depends upon many factors, such as quality of fiber and product desired, cleanness, uniformity, weight, end use and cost. Viscose rayon is used where highest quality, strength and appearance are desired in those applications where woven fabrics have been used or fabric-like qualities are preferred. Where uniformity and strength are less of a factor, cotton and rayon blends, or cotton alone, may be used. Wool has only been used in a few special applications where certain characteristics peculiar to this fiber are desired. Vinyon and cellulose acetate have been used principally as bonding fibers.

When rayon is used, it is usually the 1.5-denier, 1-9/16" or 2" staple. For lower-quality merchandise some inferior staple and rayon wastes are used, usually blended with cotton. An all-cotton nonwoven fabric seems to hold some advantages for high-pressure laminated-plastics work, while cotton-rayon blends are used chiefly in towelling.

BONDING PROCESS——The bonding process, which is a separate operation subsequent to the formation of the web and essential to the production of nonwoven fabric, is performed on auxiliary equipment, usually installed in the same production line as the web-forming machine. The type of bonding equipment used will depend upon the bonding agents and the form in which they are to be applied, that is, whether the web is to be selectively bonded, overall-bonded, impregnated, or surface-bonded.

Several of the better known nonwoven fabrics use a thermoplastic fiber, such as Vinyon or plasticized cellulose acetate, as the bonding medium. In these cases the thermoplastic fiber is blended with the basic web-forming fiber at the picker or feeder prior to the web forming operation. The amount of thermoplastic fiber used will depend upon the characteristics desired in the nonwoven fabric and the end use. It may constitute anywhere from 5 to 65% of the blend. Activation of the thermoplastic fibers to give the necessary bond is usually carried out by hot calendering. The particular type of machine, pressures, and temperatures used are largely a matter of personal choice and may require considerable experimentation before the preferred combination is arrived at.

Another method of bonding such webs is to adjust pressure and temperature to bond the thermoplastic fibers only at their crossing points so as to form a network in which the non-thermoplastic fibers are intermeshed in unbonded form and are held in place only by mechanical action and friction.

More extensively used than thermoplastic bonding fibers are liquid bonding solutions, or emulsions. These may be applied through the medium of print, dip rolls, or roller coaters, either on the outer surfaces so as to leave the interior soft, or so as to penetrate and securely bond the entire thickness of the material over any prescribed surface area.

The liquid bonding agents most commonly used are synthetic latex, such as the Geons, styrene-butadiene, water-soluble derivatives of cellulose, such as carboxy-methyl-cellulose, polyvinyl acetate or polyvinyl alcohol. Other bonding agents that may be used are cellulose xanthate, sulfuric acid, vinyl resins, starches, casein glue, or cellulose ether, such as Ceglin.

DRYING——When thermoplastic fiber-bonding agents are used, no drying is necessary other than that which may take place incidental to the bonding by some hot-pressure means, as previously mentioned. An exception to this might be activation of thermoplastic fiber by means of solvents; however, this method is both hazardous and costly and is not apt to be adopted on any wide scale.

When liquid bonding agents are used, some means of drying and/or curing is needed. This may be set up in a production line as a series of dry cans, drum dryer, hot-air oven, banks of infrared lights, or variations thereof.

DYEING & FINISHING——In some

instances it is desirable to apply certain finishes, such as water-repellent or antimildew agents, fire retardents, etc. These may be applied in conjunction with, or immediately after, the bonding solution and prior to drying, or as a separate operation after drying, and in conjunction with, or separate from, dyeing. Dyeing is usually carried out as an operation subsequent to bonding on conventional machines for dyeing flat piece goods, on padders, or on special equipment to suit a particular operation. Direct dyes are generally used, though occasionally acid dyes, vat dyes, or pigments may be utilized.

PROPERTIES

PHYSICAL CHARACTERISTICS—— As you may have gathered from this discussion, an extremely wide range of physical characteristics may be built into nonwoven materials. It is impossible in this paper, however, to cover all the various physical characteristics that are to be found in the nonwoven fabrics at present on the market. These characteristics may be controlled over an exceedingly wide range by the various methods of manufacture. An evaluation of the wear resistance of nonwoven fabrics has been covered in a report for a joint AATCC and ASTM committee *(2)*.

In presenting you with these data, I would like to emphasize that the results apply only to the particular samples tested. They represent neither the range nor limitation of physical characteristics that may be, and are being, built into nonwoven fabric so as to make it truly a versatile product worthy of consideration by all who use fibrous materials.

USES——A long list of present uses for nonwoven materials could be made in the apparel, household, and industrial fields. We shall not take the time to list all of these, but will mention only a few of which we have some examples here before us in the apparel field, such as aprons, diapers, handkerchiefs, linings, and shoulder pads. A few household applications are bedspreads, dust cloths, dishcloths, draperies, napkins and towels. In the industrial field might be mentioned backing material for plastic film and flock, filter fabrics, bags, laminated plastics, and wrapping material.

SELLING PRICE——Only a word as to prices. Nonwoven fabric at present usually ranges in price from about 4 to 30c per yard depending upon weight, fiber used, method of manufacture, etc. It is generally classed, and probably will remain, in a price bracket somewhere between that of a good grade of paper and a medium grade of woven fabric.

CONCLUSION

In conclusion, I would like to leave you with the thought that the nonwoven-fabric industry is young and is growing actively but with many pains and limitations. Nonwoven fabric will force neither woven fabric nor paper off the market, nor is it likely that it will cut a serious dent into the volume of either. It will replace certain uses for each of those materials and will develop entirely new uses for itself as a distinct product. The cost of production is at present high and frequently discouraging, though, with greater knowledge, improved technique, and higher production in individual plants this should be reduced considerably.

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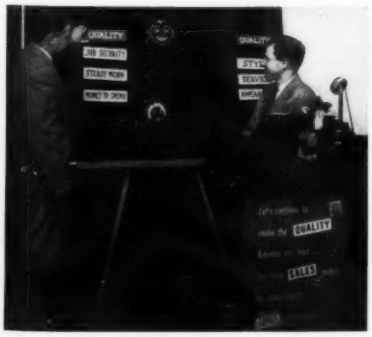
PHOTOS OF THE HUDSON-MOHAWK SECTION'S JOINT MEETING WITH MEMBERS OF THE ALBANY SECTION OF THE AMERICAN SOCIETY FOR QUALITY CONTROL ON APRIL 3 AT THE BIGELOW-SANFORD CARPET COMPANY, AMSTERDAM, N Y.

(See Meeting Report on page P364, June 9th issue)

(Right): Robert Jones Illustrates the Use of "How to Do It" Charts Employed by Bigelow Sanford to Teach Workers Correct and Best Way to Produce Quality Carpet. (L to r): Mr Jones; Irwin Smith, Surpass Chemical Co; William Childs, Behr-Manning Co; Edward Bauer, Winthrop-Stearns; John Merrill, Cluett, Peabody, Inc; Hyman Stein, Star Woolen Co; William Nelson, Ritter Chemical Co; Jack Epelberg, Cluett, Peabody.

(Additional photos on following page.)



George McGrath and Robert Jones of Bigelow Sanford

Dining in the Bigelow Sanford Cafeteria

Robert Peters, Chemical Development Supervisor, Explains Package Dyeing of Carpet Yarns

A View of Bigelow-Sanford's Dyehouse Showing Loading of Package Dye Carriers

Report of New York Section Outing

June 13, 1952
North Jersey Country Club, Wayne Township, N J

THE Annual Outing and Golf Tournament of the New York Section was held at the North Jersey Country Club, Wayne Township, N J, on Friday, June 13, 1952.

The golf tournament was won by Chris W Farrell of Bick & Co with a low gross of 73 for which he received the Association cup. Low gross for guests was won by Sam Lippincott of American Viscose Company with a score of 72. Mr Lippincott also won the prize for the shot nearest the pin. Second low gross for members was scored by Robert W Brewer of American Aniline Products, Inc, with a 77. Winner in the kickers handicap draw was Victor Moeller. Others who qualified were: Richard P Monsaert, Jr, Puritan Piece Dye Works; Walter F Lauer, Ciba Company, Inc; G Sherman Monroe, Martin Weiner Corp; Joseph B Evans, Dexter Chemical Corp; J Koenig, Tex-Chem Co; Donald E Marnon, General Dyestuff Corp; and Roy A Secord, E I du Pont de Nemours & Co, Inc.

Winners in the horseshoe pitching contest were: Ray Baker, United Piece Dye Works, 1st; Nick Stack, United Piece Dye Works, 2nd; Larry Kluger, American Aniline Products, Inc, 3rd; and Francis Habersberger, Kelco Co, 4th.

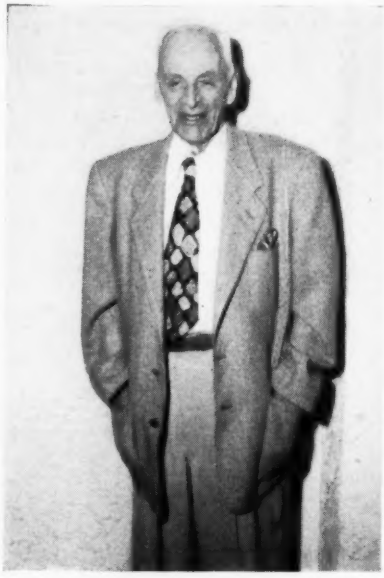
A special award of a pen and pencil set was presented to Carl Puller of Pfister Chemical Co as the oldest active dyestuff salesman in the area. The award was presented by Paul J Luck, Section Chairman, who read the following citation:

"To Carl Puller, the oldest active dyestuff salesman in the New York Section of the A A T C C:

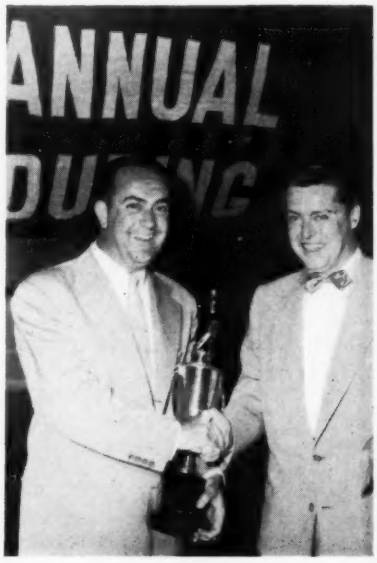
"Seventy-six years young, your agreeable personality has endorsed you to a host of friends in the textile industry.

"It is their hope that this little gift may prove of practical value, but beyond this, may it be a daily reminder that you are always in their thoughts.

"Long life and health to Carl Puller!"



Carl Puller



Chris W Farrell (left) receives Low Gross Trophy from Outing Chairman Ernest J Gsell

The Martin Weiner Fashion Plate Award for the most colorful print pattern on a sport shirt went to Herman G Sandler of Interchemical Corp. Judges for the contest

1952 New York Section Outing Committee
(L to r): R F Holoch, C W Farrell, E J Gsell, F L Gonnerman

were Chris W Farrell; Clark A Heydon, United Piece Dye Works; and P J Wood of Royce Chemical Co. The award was presented by Charles Minoff of the donor company.

The committee for the outing consisted of Ernest J Gsell, Gsell Textile Printing Company, Chairman; Chris W Farrell, golf; Robert F Holoch, National Aniline Division, Allied Chemical & Dye Corp, horseshoes; and Fred L Gonnerman, Carbic Color & Chemical Co, prizes. It was announced that Mr Farrell would be chairman for the 1953 outing.

The attendance was 350.

Respectfully submitted
NORMAN A JOHNSON
Secretary

Committee members and officers of the Philadelphia Section at their recent outing at the Lulu Temple North Hills Country Club. Seated, left to right, J L Fancourt of W F Fancourt Co; E C Diehl, (Chairman), Ankokas Dyeing and Processing Co; F V Traut, (Treasurer), Globe Dye Works, and T J Scanlan, (Secretary), Calco Chemical Div. Back row, J W MacNeil (Publicity Chairman), American Aniline Products, Inc; F E Nevins, Ciba Co; E W Empting, General Dyestuff Corp, and C T Anderson of the Ciba Co.

EMPLOYMENT REGISTER

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute. Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the Secretary.

52-15

Education: BS, Chemical Engineering.
Experience: Textile chemist, technical service and research.

Age: 35; married; references; technical service, advertising-sales or liaison work desired, near New York if possible.

6-23, 7-7

Over 400 Attend Philadelphia Section Outing

THE Philadelphia Section held its Annual Outing at the Lulu Temple Country Club, North Hills, Pennsylvania, on Friday, June 13th, with over 406 members and guests in attendance.

Golfing honors for members went to Thomas C Jordan, W H & F Jordan Chemical Company, Inc with a low gross of 71. Top honors for guests went to J Mathewson, Carbide & Carbon Chemicals Co, with a low gross of 78. Mr Jordan was awarded the Annual Golf Trophy, golf bag and also an individual golf trophy donated by the Philadelphia Section. Mr Mathewson received a pair of golf shoes as his prize.

E Smith, Nyanza Color & Chemical Co, won second prize with a low gross for members of 73. J Chuck, Charles J Haas Co, last year's winner, placed third with a low gross of 76. R Hansen, *Textile Industries,* placed fourth with a low gross of 76 and R C Brown, E I duPont de Nemours & Co, Inc, placed fifth with 77.

M Crystal, Thermco Products Inc, was second prize winner for guests with a low gross of 80.

R Kroener, Kohout & Fidler, was third with a low gross score of 81.

Low net scores were divided into two classifications—Class "A" with scores of (100—) and Class "B" with scores of (100+).

Class "A" (100—)		
1st	Art Snider	79-70
2nd	Art Gordon	83-70
3rd	R. Jaegers	81-71
4th	C. E. Seel	89-71
5th	J. Jordan	78-72
6th	J. Phillips	81-72
7th	A. S. Kohout	83-72
8th	G. Rosset	91-72½
9th	J. M. Pearce	93-72½
10th	T. A. Mahan	85-73
11th	J. L. Palett	92-73
12th	A. E. Bride	96-73
13th	J. Corea	96-73
14th	J. R. McCullough	91-73
15th	F. M. Tolin	80-74
Class "B" (100+)		
	R. E. Nickerson	101-72½
	H. C. Harvey	102-73
	J. W. Brown	108-73
	Ralph Nebuling	126-73
	J. Ritz	101-73½
	C. J. McLaughlin	125-74
	Dave Leshin	126-74
	Bill Kirk	135-74
	W. E. Filler	106-75
	J. E. Conway	106-75
	T. Poole	107-75
	G. McCullough	117-75
	E. J. Glazer	117-75
	W. Diamond	121-75
	Jack Hoffner	101-75½

H Furlong won the close ball contest on the 12th hole coming 9 inches from the cup. A S Kohout, Kohout & Fidler, placed second 20½ inches from the cup.

The putting contest was won by A Franks, Alex C Ferguson Co with a score of 31. M Schiff, Thermco Products, Inc, was second with a score of 33.

First honors in the quoits contest went the team of C Seibert, Scholler Bros, and E Smith, Nyanza Color & Chemical Co Inc. Second prize was won by the team of A Raimo, John Campbell Co, and A Weikel, Interstate Hosiery Mills.

W Littlewood, G J Littlewood & Sons, M Bartholomew, Florence Thread Co, and J Lord, Waverly Dyeing and Finishing Co, placed first, second and third in the dart contest.

The golf irons raffle was won by C E Hunter, H W Butterworth & Sons Co.

James Peirce, Dow Chemical Co, won the one hundred dollar savings bond drawing and John McChesney, Leatex Chemical Co, won the twenty-five dollar savings bond drawing.

The partners, Elmer Zeurner, Sterling Dye & Finishing Co, and Charles A Schmettler, Scholler Bros, took first place in the Pinochle Contest. Thomas Halst and Jasper Murphy, Laurel Soap Co, finished second.

Numerous other prizes for golf, quoits, darts and cards were awarded and over 50 door prizes were won by members and guests.

An excellent floor show followed dinner.

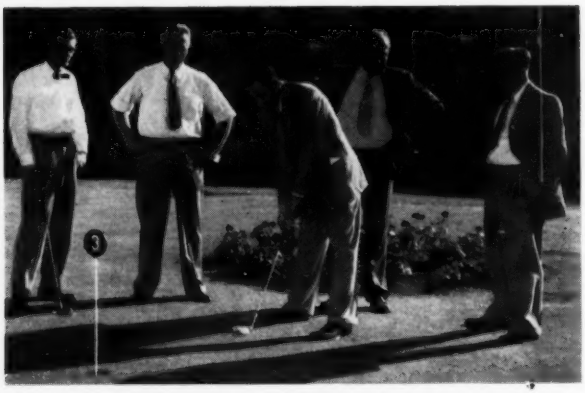
The outing was under the Chairmanship of John L Fancourt, W F Fancourt & Co.

PHILADELPHIA SECTION OUTING PHOTOS (1952)

(L to r): D W Robinson, Alco Oil & Chemical Co; G J Littlewood III, G J Littlewood & Sons Co; C A Seibert, Scholler Bros, Inc; J L Fancourt, W F Fancourt Co.

(L to r): G J Moffitt, American Aniline Products, Inc; C Duddy, Kaufman Plush Co; G McCulloch, American Cyanamid Co; R Jaegers, Schuykill Haven Bleach & Dye Works; J Bamford, Schuykill Haven Bleach & Dye Works; J Lord, Waverly Dyeing & Proc Co; J Hulton, W E Hulton Dyeing Co.

(L to r): J L Fancourt, W L Fancourt Co; R Hill, E W Empting and P Pernice, General Dyestuff Corp.



On the putting green at the Philadelphia outing: (l to r) J Bowdler, Arnold Hoffman Co; D MacGregor, D Grant, K Harper and R Hunt, all of Franklin Process Co.

Patents———

(Concluded from Page 416)

Other examples refer to analogous printing and dyeing operations with various basic dyes and also one type of acid dye (Xylene Blue AS); claims protect the use of basic dyes only.

Among other references cited by the Patent Office:

Ger P 485,894 (I G Farbenind A G/1929): basic or acid dyes containing one or more sulfonic acid groups are known to form light-resistant lakes with phosphotungstic or -molybdic acids or salts thereof. These lakes are obtained in situ on the fiber by preparing the goods with one of these salts and printing or dyeing with basic dyes (cf U S Pat 1,912,878 above).

Brit P 522,591 (Brit Celanese/1940): colored lakes are formed from insoluble anthraquinone dyes (used for coloring acetate of cellulose) and phosphomolybdic acid or salts thereof. (This patent principally protects lake formation with the dye but not in aftertreatment).

Brit P 566,258 (Calico Printers Ass'n/1944): insoluble color lakes are fixed on fibers by printing a basic dye and alkali phosphomolybdate or tungstate, steaming to effect penetration, and finally insolubilizing the lake by an acid treatment, preferably by steaming in the presence of acid vapors.

PRINTING DIRECT OR ACID DYES——— Fixation with Cyanamide-Formaldehyde D, 2, 03

Brit P 668,421

(Courtaulds Ltd——Cluley, et al——March 19, 1952)

The co-pending Brit P 652,985 (cf *Am Dyestuff Reptr 40,* 747 (1951) describes a process for printing acid or direct dyes using a water insoluble condensate of cyanamide plus formaldehyde, adding a potentially acid salt, such as ammonium formate, and steaming. The purpose of the acid developed in the steaming operation is to solubilize the water insoluble condensate to effect penetration of the resin solution and probably to transform the solubilized resin into the insoluble state by subsequent drying.

The present modification of the aforementioned process consists of printing a water soluble dyestuff, a thickening agent and a water insoluble cyanamide-formaldehyde condensate as above while using an organic (preferably bound to a strong base) as a solubilizing agent.

Example: a paste is prepared from Chlorazol Sky Blue FF, urea (as a dispersing agent), copper acetate (as a light fastness improving agent), sodium lactate plus lactic acid, and a glycerol paste containing the insoluble resin condensate; the whole composition thickened with gum tragacanth. The prints are dried and steamed for 45 min at 100-102° C, then washed with water or lukewarm soap solution. The thickener is removed and no substantial bleeding off occurs. Washing with dilute ammonia is recommended when acid sensitive dyes are used.

PRINTING ACETATE OF CELLULOSE——— Condensates of Ricinoleic Acid and Ethylene Oxide D, 2, 07

Brit P 664,646

(Imperial Chemical Industries——Batty, et al——Jan 9, 1952)

Dispersed, water-insoluble dyes of the azo and anthraquinone series are generally used for printing nylon as well as acetate of cellulose. It has been observed that brighter shades can be obtained by adding condensates of ricinoleic acid and/or bodied ricinoleic acid with ethylene oxide.

Ricinoleic acid may be bodied according to standard methods by heating in vacuo. Commercial castor oil containing a certain amount of bodied ricinoleic acid may be employed. This product is condensed with 5-17 mols (preferably 11.6 mols) ethylene oxide. About 1 per cent of this condensate is incorporated in the printing paste.

Example: an aqueous suspension of the dye obtained by coupling diazotized p-nitraniline with di (β-hydroxyethyl) aniline is dispersed in a tragacanth thickener. Some "Ludigol" and 4% of a 25% solution of the condensate (described above) are added. After printing and steaming takes place for 30 minutes, a brilliant red is obtained.

Printing acetate dyes with ricinoleic acid soaps is known per se, as is the adding of nonionic compounds or Ludigol to these pastes (cf *Melliand Textilberichte,* 1926, 354, Schneevogt). The innovation is in the application of a specific ricinoleic acid——ethylene oxide compound prepared under controlled conditions.

ABSTRACTS

Fabric Shrinkage and Launderability

C Wills, *Canadian Textile J* 69, 55-7, February 15, 1952.

Although we may consider that, in the main, the mechanically shrunk cotton and linen fabrics and the stabilized rayons being produced today have overcome many of our shrinkage problems, the fact remains that we do not produce by these processes a fabric from which a garment can be made which will retain its original dimensions after repeated launderings.

Even the fully shrunk shirt (so-called) will give indication that some shrinkage still persists after five or six visits to the laundry by the tightening of its collar.

This condition is not indicated in the standard washing test procedure for the testing of Sanforized fabric after the shrinking process. Also in spun rayons, the stamp of approval is given on an initial test, not on a series of tests to approximate repeat laundering.

Other points to be considered are the construction of the fabric and its end uses. For instance, a broadcloth shirt, which is more often than not in a mercerized finish, will withstand 25 to 30 launderings with a shrinkage loss of approximately 2.25 per cent beyond the initial fabric shrinkage, while an 8-oz drill used for overalls will under controlled conditions stand up to approximately the same number of launderings, but with an altogether different progressive shrinkage until it finally stabilizes around 8 per cent.

After discussing the physicochemical properties of the cotton fiber, the author describes experiments made on a cotton drill in an attempt to reduce this progressive laundry shrinkage. It was found that mercerization reduced the figure from 8 to a little over 5 per cent, and that further treatment with resins, in particular with melamine formaldehyde, reduced it still further to about 3 per cent. He comments, however, that the resin was very gradually removed by the successive launderings.

Reference is made to the new type of resin application patented by Dan River Mills, using starch methylol-acetone methylol copolymer, which is said to give better stability, crease resistance, and permanency of finish.

The author also discusses the shrinkage of rayon fabrics and various attempts at stabilization, which may be summarized as follows:

(1) Introduction of formaldehyde cross-

linkages within the fiber (e g, by means of Glyoxal).

(2) Resin treatments.

(3) Replacement or substitution of hydroxyl groups in the cellulose molecule by less hydrophilic radicals.

For resin application, he suggests that an application of 12.5 per cent urea or melamine resin solution will give good stabilization to a summer weight suiting. An addition of Zelan or Norane to the resin, he states, should improve the stabilization still further.

He believes that further research must be done before the problem of shrinkage control is finally solved.

Partial Acetylation of Cotton

A S Cooper, S T Voorhies, Jr, E M Buras, Jr, and C F Goldthwait, *Textile Industries* 116, No 1, 97-102, 194-5 (1952).

Cotton substituted chemically, as by partial acetylation, becomes virtually a new fiber. Although it does not change in appearance or in general textile qualities, new properties are imparted by partial acetylation which make this modified cotton of definite interest to textile manufacturers. These properties include high resistance to mildew and microbiological rotting; resistance to the effects of high temperatures or continued heat; low moisture regain and high electrical resistance, as compared with untreated cotton, and resistance to coloring with many direct dyes.

Progress has been made at the Southern Regional Research Laboratory in acetylating cotton, and raw stock, yarn, and cloth can be treated by batch methods on standard textile equipment. Also, yarn and cloth have been acetylated on a pilot-plant scale by a continuous method now being developed.

The property now arousing most interest is resistance to heat, as in the pad covers for laundry "hot head" presses. Acetylated sheeting has lasted four to five times as long in this severe service as the usual cotton canvas covers.

A promising use already well tested in commercial units is for bags to hold zeolite in household water-softening systems. Partially acetylated bags remained intact during a normal year's service, whereas ordinary bags developed holes in a month or two.

Acetylated fishing twines were found to last two to three times as long as twines protected by tar, and at least eight to ten times as long as untreated twines.

The authors give complete instructions for the acetylation process. The cotton must first be purified by the removal of all noncellulosic material. It is "activated" by soaking for at least four hours at room temperature in glacial acetic acid. The acid is then drained or squeezed from the cotton to be used again. The chemicals employed for the acetylation are commercial glacial acetic acid, acetic anhydride, and small amounts of a catalyst, perchloric acid, conveniently obtained at 60 per cent solution. The reaction mixture must be continuously cooled to prevent any harmful increase in the rate of reaction from rise of temperature.

The treated materials are washed in cold water until most of the acids are removed. Hot water may then be used, or the acids may be neutralized with ammonia.

The extent of acetylation can be determined by the usual chemical analysis by saponification with excess sodium hydroxide and back titration with acid. Such a determination, however, does not indicate whether the cotton is uniformly acetylated sufficiently within the sample tested to have the desired properties. However, uniformity within a sample of any desired size can be determined qualitatively by a dyeing test. A combination of a direct dye, Chlorantine Fast Blue 3RLL, and an acetate dye, Celliton Fast Yellow RRA, has been found suitable. In this test the unacetylated cotton dyes a full reddish blue and well-acetylated cotton a full yellow. Cotton not well acetylated looks green.

The properties of partially acetylated cotton, and the moderate cost of production (about one-half of the cost of grey cotton cloth), in view of the exceptionally long life of the treated cotton under conditions where ordinary cotton fails relatively quickly, make it a promising item for commercial manufacture.

Sizing of Spun Nylon Yarns with Various Agents

J H Langston, *Textile Research J* 22, 111-16, February, 1952.

This article describes a large number of trials conducted at Clemson College in an attempt to find a suitable sizing material for spun nylon yarn. The author points out that in addition to the general requirements of any size mixture, the most important criterion of a satisfactory size for spun nylon is that it hold down the ends of the staple fibers in the yarn, which otherwise tend to work to the surface during the abrasive action of the weaving process, causing fuzzing of the yarn surface and consequent tangling of the warp ends. From this standpoint, starch, polystyrene, and dimethylol urea were found to be completely ineffective as sizing agents. Gluconic acid offered some promise, but was too hygroscopic for practical use.

As a result of these trials the author draws the following conclusions:

(1) Of the several potential sizing agents for spun nylon which have been evaluated, the most promising appear to be polymethacrylic acid, polyacrylic acid, and polyvinyl alcohol.

(2) The sizing efficiency of all these materials is increased by the addition of 1%-3% of sodium alginate to the size mixture.

(3) A size mixture containing 4.0% of polymethacrylic acid and 1.8% of sodium alginate has been used with good results in the slashing and weaving of an 18/1 spun nylon warp.

The author believes that further and more complete investigation into the use of alginate-containing spun nylon size mixtures should be carried out.—WHC

Dyeing Dynel Stock, Yarn, Hose

T A Feild, Jr, *Textile Inds* 116, 139, 142, April, 1952.

When dyeing dynel, the dyer has latitude in dyes and procedures. It should be remembered, however, that the fiber is thermoplastic.

Acetate Dyes. Use a minimum amount of dispersant. Dynel will blush in boiling water or at 205°F; to avoid this, add sodium sulfate midway during the dyeing operation.

Acid Dyes. For pastel shades, apply the dye from an acid bath at the boil. No other assistants are required. The fiber should be thoroughly wet out first. For medium shades, small amounts of copper sulfate with zinc formaldehyde sulfoxylate (to reduce the copper) are added to the bath at room temperature; the dye is then added, and dyeing conducted at the boil for 1 hour. The pH of the bath should be 5. For heavy shades, a swelling agent, p-phenylphenol, is added at the start. The bath is adjusted to pH 6 with sulfuric acid. The copper sulfate, reducing agent, and dye are then added as in the case of medium shades. Blushing can be prevented by addition of sodium sulfate.

Acetate-Acid Dye Mixtures. Dye at the boil with both types of dye present, usually without a swelling agent. After 20 minutes, adjust the bath to pH 6 and drip into the boiling bath a cold mixture of copper sulfate and reducing agent to exhaust the acid dye.

Acetate dyes have excellent leveling qualities, very good wash fastness, and excellent light fastness. Acid dyes (using the copper technique) have outstanding wash and light fastness.

Stock Dyeing. When dyeing in closed machines, use the minimum amount of agents that tend to foam. If necessary, add an antifoam agent.

Hosiery Dyeing. Acetate dyes are usually employed. Dye at 205-210°F for 30 minutes, add sodium sulfate and continue for 1 hour.

Dynel is seldom scoured for light shades, but should be scoured with soap or an anionic detergent for dark shades—WHC

NOTE: The full text of this paper will appear in the AATCC *Proceedings* shortly.—ED.

Dyeing Acetate with Naphthols

W R Ivey, *Textile Inds.* 116, 191, April, 1952.

A practical method for package dyeing of acetate yarns with Naphthol colors has been developed to make possible the production of yarns with greater wash fastness than that provided by the acetate dyes.

The naphthol is dissolved with denatured alcohol, caustic soda, and a nonionic dispersing agent. The machine is filled with water, a synthetic detergent is added, and the Naphthol solution is poured in and well circulated. The yarn packages are entered dry and circulation is continued, the temperature being gradually raised to 120°F. The prepared color base is added, the temperature is raised to 160°F, and monosodium phosphate is added. After one-half hour the packages are rinsed with cold water containing 20 per cent of salt, then with water alone.

The machine is again filled, the temperature is raised to 120° F, and sodium nitrite and hydrochloric acid are added. The temperature is further raised to 160° F and diazotization is continued for 15 minutes. The packages are then washed with soap and soda at the boil.

By the use of this process it is possible to obtain light fastness of 40 to 100 hours, depending on the Naphthol and base employed, and excellent wash fastness.—WHC

NOTE: The full text of this paper will appear in the AATCC *Proceedings* shortly.—ED.

The Clothing Fire Hazard

"Technologist," *Textile Mercury and Argus* 126, 597-600, 686-90, 736-41, April 4, 18, and 25, 1952

It is quite a simple matter to render a fabric flameproof, but almost all flameproofing treatments have the serious disadvantage of harming the fabric in one way or another and lowering its quality.

Among the many requirements of a flameproofing finish are the following: (1) the treatment must not cause dermatitis; (2) it must not reduce the tensile strength or useful life of a fabric; (3) it must not produce stiffness or a tendency to crease and drape badly; (4) it must not radically change the dye affinity of the textile fibers or set up a slow deterioration; (5) it must not interfere with any treatment applied to render a fabric fast to washing, laundering, and weathering.

The author discusses first the nonpermanent fire-retarding agents, in which category he lists borax, boric acid, diammonium phosphate, ammonium sulfate, ammonium sulfamate, antimony oxychloride, zinc chloride, and others. Some of these products apparently act by melting in the flame and forming a sort of protective film. on the fibers. Others appear to depend on their ability to form acid radicles when heated, which are capable of reacting with the cellulose fiber to form esters.

The author next describes Du Pont's "Erifon" flame-retardant, which consists of a combination of titanyl chloride and antimony trichloride. He states that titanyl chloride is able to combine chemically with cellulose to form a stable insoluble compound, and that while cotton treated with the titanyl chloride alone acquires a fair degree of resistance to flaming, this effect can be much enhanced by the presence of antimony oxide.

It has been found that some types of Erifon-treated fabrics can be laundered 100 times with soap at 140°F without appreciable loss of their flame-retarding properties. Caustic alkalis, however, applied at high temperatures can have a definitely harmful effect.

The author next discusses the Bancroft process, in which the cotton fabric is impregnated with a mixture of urea and phosphoric acid and cured at 125-175°C. The phosphoric acid is said to combine with the cellulose, and the urea acts as a basic buffering agent to protect the fiber from degradation. The phosphorus is so firmly held by the cellulose that it will stand boiling with 1 per cent caustic soda solution, it is said. However, unless the Bancroft process is carefully controlled, there may be a serious loss of strength by the cotton fabric.

Finally the author describes another type of permanent treatment, involving the use of antimony oxide and a chlorinated body, such as chlorinated paraffin. By itself antimony oxide is a relatively poor flameproofing substance, but in the presence of a chlorinated compound it can be very effective.—WHC

Acrilan Acrylic Fiber Dyeing and Finishing

J A Woodruff, *Canadian Textile J* 69, 58-61, May 23, 1952.

Acrilan is the acrylic fiber manufactured by the Chemstrand Corp. Like the fiber, this company is new to the textile trade, since it was formed in 1949 as a jointly owned subsidiary of the American Viscose Corp and the Monsanto Chemical Co. At the present time the limited production of fiber is located in a pilot plant in Marcus Hook, Pa.

Acrilan fiber is made of a modified polyacrylonitrile resin spun into filaments in a liquid spinning process. It is not marketed as a continuous filament but in the form of crimped staple fiber of either three or five deniers per filament. Acrylonitrile is not swollen by water, and therefore Acrilan is classified as a hydrophobic fiber.

The fiber is thermoplastic, with a shrinkage of 5 per cent in dry air at 350°F and a softening point over 400°F. To the dyer and finisher, this means that some degree of heat setting will be of importance.

Acrilan can be colored experimentally by nearly all dyes if the proper conditions are used. The easiest to apply are the acetate dyes which dye quite readily at 200°F and upwards. Unfortunately, the average wet fastness and light fastness of these dyes is poor and they have therefore found only limited use.

Vat dyes are now receiving attention in the laboratory and without doubt will find commercial application for some uses in the not too distant future.

Those dyes normally used for dyeing wool are also good for dyeing Acrilan. The fastness properties of the dyeings vary widely, and considerable laboratory selection has been done in order to obtain those dyes having the best combination of properties. In the remainder of the paper all dyes mentioned are wool type dyes.

Time is a very important function of Acrilan fiber dyeing. In order to obtain well penetrated dyeings, it is necessary to use approximately two hours time with the temperature at 200-205°F, and with acid dyes a concentration of 10 per cent sulfuric acid on the weight of the goods. If the temperature of dyeing is raised above 212°F, the apparent rate of diffusion into the fiber is greatly increased, and at 235-240°F very short times of dyeing are possible. Little dyeing takes place below 160°F.

In dyeing Acrilan with wool dyes, one must use considerable acid in order to promote exhaustion and aid in the diffusion of the dye into the center of the fibers. The rate of diffusion is speeded up tremendously with increasing acid content. In most cases it has been found useful to use sulfuric acid. With certain dyes, however, nitric acid has a strong effect on the dyeing properties when used in equivalent percentages to those used with sulfuric acid.

Detailed instructions are given by the author for stock, top, yarn, and piece dyeing.

In finishing Acrilan fabrics it is necessary to be careful with prolonged heating in dry heat, such as an extended curing cycle; beyond five minutes at 315° F, for example, yellowing of the fiber will take place.

It was brought out in the discussion period that at present there is no method of commercially bleaching Acrilan.—WHC

NEWS

• National Aniline Plans to Build New Aniline Plant

The National Aniline Division of Allied Chemical & Dye Corporation proposes to build a new chemical plant for manufacture of nitrobenzene and aniline by newly-developed continuous processes on a site south of Moundsville, West Virginia, it is learned. The site is adjacent to a new plant of the Solvay Process Division now in the course of construction which will manufacture chlorine and caustic soda using local salt deposits. The new catalytic process for aniline will use hydrogen generated in the Solvay operation.

Construction of National's new plant will reportedly start at an early date with completion projected in 1953; work will be directed by the Division's Engineering Manager, F J Krueger. Because of the plant's importance to the defense effort, the Defense Production Administration has awarded Certificate of Necessity covering 60% of the cost of the facilities estimated to exceed $2,300,000, it is announced.

The Division now manufactures dyestuffs, detergents, pharmaceuticals, intermediates and related chemicals at its Buffalo, N Y, Plant.

• Donald Named President of New Celanese Organization

Celanese Corporation of America, New York, has announced the formation of a new global organization, Celanese Central, S A, which reportedly will provide closer liaison with the activities of foreign affiliates of the company in all countries except Canada. Kenneth G Donald has been named president of the new organization.

Celanese has stated that during the past few years the size and scope of its activities have substantially expanded with the result that management has been faced with constantly increasing duties. It added that interests in other countries have become so large and diverse that a continuing liaison with these countries is needed, particularly at this time when the difficulties of foreign trade are becoming more pronounced.

In Canada, the firm has already established Canadian Chemical and Cellulose Co, Ltd. It has four foreign affiliates in Mexico, one in Colombia and one in Venezuela.

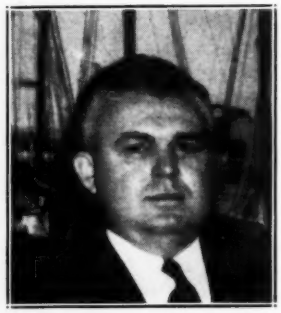
• Aquex Names Southeastern Distributor

The Aquex Development & Sales Corp, Whippany, N J, has announced that the Moreland Chemical Co, Inc, 314 West Henry Street, Spartanburg, S C, will be district representatives for the sale of its new resins for textile finishing.

E W Sweet, Vice President and General Manager of Aquex, points out that production runs under actual mill conditions have demonstrated that these new finishing resins — called Aquex BNW and Aquex BNWC — reduce residual shrinkage to a minimum and greatly improve abrasion resistance.

Increased acceptance of the resins by the textile trade has merited the appointment of Moreland as the Southeastern representative, according to Mr Sweet.

OBITUARY



W Naef

WILLIAM NAEF

WILLIAM NAEF, textile colorist with the Kelco Company, New York, for the past two-and-a-half years, was killed on June 2nd at Dover, N J, when his car went through the guardrail of a Route 6 overpass and plunged to the tracks of the Central Railroad of New Jersey, 40 feet below. He was alone in the car.

Mr Naef started his career in the wet-processing field with General Dyestuff Corporation in 1930. He later went with Concordia Textile Printing Co previous to joining Kelco.

He is survived by his widow, Libera, a son, Robert, and a daughter, Marilyn.

R C Sarfaty

• Sarfaty to Head New Cyanamid Department

American Cyanamid Company has formed a Sales Service Department for its Industrial Chemicals Division. A J Campbell, General Manager of the Division, explained that the move is in conjunction with a reorganization program, which has already consolidated various functions in the district offices. The new Department will reportedly provide trade records, sales analysis, and complete order processing for the Division's main office in New York.

Appointed manager of the new Department was R C Sarfaty, who formerly served as a product supervisor for Industrial's Heavy Chemicals Department. He joined Cyanamid in 1945.

E O Heydt will be in charge of the Trade Record Section, and A L Wycoff, Jr will supervise the Order Processing Section.

• DRM Sales Office in Montreal Opened

The opening of a Dan River Mills Canadian sales office at 1214 University Tower Building, Montreal, was recently announced by Russell B Newton, President of Dan River Mills, Incorporated, Danville, Virginia.

Frank L MacAskill will head the new office, it was disclosed.

• AFD Names Snell as Official Lab

The appointment of Foster D Snell, Inc, New York firm of consulting chemists and chemical engineers, as official laboratory of the Association of Food Distributors, Inc, has been announced by T R Schoonmaker, executive secretary of the association.

• ISO Notes Progress in Measuring Shrinkage

A group of 33 textile experts from twelve nations, working at Columbia University as a sub-committee of the International Organization for Standardization (ISO), has reported marked progress on development of a set of international standards and specifications for measuring the shrinkage of textiles.

W D Appel, chief, Textile Section, National Bureau of Standards, chairmanned the meetings and headed the eleven-man American delegation. Mr Appel has been active in textile standards work of the American Standards Association, which is the U S member of the ISO and acted as host to the delegates from 25 nations which met recently in two-week sessions at Columbia.

Basic for the textile committee's discussion was a standard prepared and submitted by the American Standards Association for the U S "Shrinkage in Laundering of Woven Cotton, Linen and Cotton-Linen Fabrics." The following action is reported to have been taken on the U S proposal.

1. The French asked for a broadening of the scope to include hemp and mixture of synthetic fabric fibers. The proposal was turned down, but the committee will give later consideration to the matter of hemp.

2. The committee agreed to eliminate preliminary drying prior to extracting and pressing.

3. The committee agreed that wash wheels shall be not less than 18 in in diameter, have 1 to 3 baffles, and rotate at a speed which will lift and drop the materials (peripheral speed of 176 feet per minute is satisfactory), reversing every 1—8 revolutions. It was agreed that there should be facilities for rapid heating of the washing liquor (live steam, gas, electricity are satisfactory).

4. Liquor to air dry fabric ratio: the goods shall be covered with liquor but in no case shall the liquor to air dry fabric weight exceed a ratio of 20 to 1.

5. Air dry fabric to cage volume ratio was agreed on at 2.8 to 3.2 pounds per cubic foot.

6. An extractor shall be used, or substitute methods which will reduce moisture retention to 50 to 60 per cent based on air dry weight of material.

7. Pressing equipment: the committee agreed on a preference for flat bed press with a hand iron as alternate.

Since the pressure exerted on the hand iron is important rather than the weight of the iron, the weight factor was eliminated. Members will check individual practices to see if suggestion of 30 to 35 grams per square centimeter pressure is satisfactory.

8. Measuring scale: the committee agreed on use of a graduated rigid measuring rule for laying out marked distances before and after laundering.

9. Test specimen: the committee agreed on a minimum size specimen of 22 by 22 inches. There is to be no maximum size limitation. Specimen shall have no marks closer than two inches to any edge and marks shall be staggered if possible.

The U S delegation agreed to attempt revisions of the ASTM specifications, federal specifications, and American Standards as required by these agreements.

10. The committee agreed on following order of operations. Add approximately 2 grams per liter of sodium carbonate; raise temperature of liquor and maintain at 95 degrees centrigrade minimum; add sufficient soap chips to give good running suds. (If more than 5 grams per liter of soap is used, the amount and reason for use of excess shall be reported). It was agreed that standard specifications for soap modified as needed for international use should be included in the body of the standard.

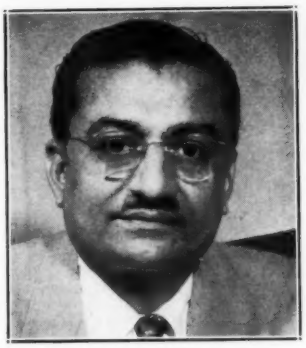
11. Time of washing and rinsing cycle. The delegations were unable to agree on the duration or need for repetition of tests until ultimate shrinkage is obtained. The U S delegation favored the test as outlined in its proposal involving a 40-minute washing cycle and a total duration of test of 60 minutes. The United Kingdom favored a 60 minute washing cycle with a total duration of test of 90 minutes. The French favored shorter washing runs repeated five times, with a total duration of 200 minutes.

12. The committee agreed that the report shall contain the average shrinkage or gain values in the warp direction and filling direction separately, as a percentage of the dimension before laundering, but not beyond the first decimal place, as shrinkage, or gain, of the specimen in the test.

The U S delegation, as secretariat of the subcommittee, agreed to redraft the proposed standards, including agreements reached, and to send the revision to each member, together with statement covering the unresolved matters, concerning which each member body will continue its investigations.

The foreign nations represented were: Belgium, Brazil, Denmark, France, Germany, India, Italy, Spain, Sweden, Switzerland, and the United Kingdom.

In addition to the chairman, the U S delegation consisted of: J Robert Bonnar, American Association of Textile Chemists and Colorists; Larus R Burgess, Cone Export and Commission Co; D E Douty, chairman and G R Turner, both of U S Testing Co; Julius B Goldberg, director of research, J P Stevens & Co; Fred Kern, F P Maupai Dyeing Co; Leonard S Little, Better Fabrics Testing Bureau and chairman of the General Research Committee, AATCC; J A Truitt and Dr C O Werner, both of the American Viscose Co; and John Warner, Cluett, Peabody & Co.



J H Doshi

• Bombay Textile Official Visits Hilton-Davis Plant

J H Doshi, director of Amritlal and Company, Bombay, India, sales representative in that country of The Hilton-Davis Chemical Company, has completed a one-week visit to consult on policy matters with officials of the Cincinnati firm. The Indian concern handles sales of Hilton-Davis naphthols, bases, stable salts, Spectrolenes and pigments.

Amritlal and Co is said to be the largest independent distributor of textile dyestuffs in India. In addition to discussing a coordinated sales program with Nelson S Knaggs, vice-president in charge of sales, and James F Thompson, general manager, Mr Doshi studied latest developments in the research and production of textile dyes at the Hilton-Davis plant.

The Bombay headquarters of Amritlal consists of research laboratories, mixing rooms, warehouses and administrative offices. The firm also maintains sales branches in all of India's major textile areas.

• Calco Relocates Hub Sales Office

The Dyestuff Division, Calco Chemical Division, American Cyanamid Company recently announced the location of their Boston sales office, laboratories, and warehouse at 1055 Commonwealth Ave, Boston 15, Mass. The new location will be headquarters for the Department's Northern New England personnel.

• FRL Represented by Williams in Scotland

Dr Simon Williams represented the the Fabric Research Laboratories, Inc, of Boston, Mass, and the Office of Naval Research at the International Conference on Fiber Blending, which was held in Edinburgh, Scotland from June 2-6, under the auspices of The Textile Institute. Dr Williams is currently visiting manufacturers and research organizations in England and on the Continent.

• Solvay Executive Sales Offices Removed

The executive sales offices of the Solvay Process Division, Allied Chemical & Dye Corporation were removed on June 9th from 40 Rector Street to 61 Broadway, New York 6, N Y.

• Finishing System Promises Better Dulling

Amalgamated Chemical Corporation, Philadelphia, has reportedly developed a new system for finishing women's nylon hosiery which is said to combine a resin finish, a dulling agent, an exhausting agent and a wax lubricant all applied in one bath after the dye is dropped.

Excellent dulling results achieved with this new "Filaseal System" are said to give promise of new life for hosiery sales. By varying the components of the system, the finish reportedly can be modified to be either soft or hard, with or without scroop, and firm or elastic, depending upon the requirements of the individual hosiery mill.

"We feel certain that we can develop a finish to meet any hosiery manufacturer's requirements", advises Harold B Dohner, Amalgamated president. "Laboratory tests have shown that samples finished with the new Filaseal System have better dulling, increased snag and pull resistance and have retained their shape throughout handling operations".

• Dedication of New Library Highlights LTI Alumni Day

Dedication ceremonies for the Lowell Textile Institute Alumni Memorial Library highlighted a program of events on June 7 at the Institute's annual Alumni Day.

Presiding at the ceremonies, which were held in the Stevens Reading Room of the new building, was Professor William G Chace, director of the library. The opening address was made by Keyes D Metcalf, director of libraries at Harvard University.

Dr G Nathan Reed, LTI's Dean of Faculty, and Douglas F Grady, '52, Student Council president, teamed to dedicate the library and Ralph K Hubbard '11, President of the Alumni Association, made the presentation to Martin J Lydon, LTI President.

The library, built at a cost of more than $300,000, was made possible through funds contributed by alumni and industry.

Another feature of Alumni Day was the awarding of honors for outstanding service to the Association to Ralph K Hubbard, Packard Mills, Inc, Sydney P Munroe, Hesslein and Company, and James G Pottinger, Reliance Manufacturing Co. Honorary memberships were presented to Dr Walter J Hamburger, Fabric Research Laboratories, Inc and Visiting Professor at LTI, and Nathaniel M Mitchell, textile engineering consultant.

June 9th saw the presentation of 127 Master's and Bachelor's degrees in textile chemistry, manufacturing, and engineering, as well as five honorary degrees at Commencement ceremonies in Lowell.

Degrees of Master of Science were presented to General Georges F Doriot, President of the American Research and Development Company of Boston and Commencement speaker; William D Appel, Chief of the Textile Section of the National Bureau of Standards; Francis P Madden, Boston fabrics distributor and a long time member of the Institute's Board of Trustees; Harry Reimer, Editor of the *Daily News Record;* and Laurence F Whittemore, President of the Brown Paper Company, Berlin, N H, and the Brown Corporation, La Tuque, Quebec.

• 51st PTI Alumni Reunion

The Alumni Association of the Philadelphia Textile Institute celebrated its 51st Anniversary with a three-day program early last month.

An open house at the Institute was the initial feature on the program on June 5th. On June 6th the Manufacturers Golf and Country Club (Oreland, Pa) was the site of the annual Outing and Reunion, which featured golf, softball, swimming, tennis, etc. Colonel C F H Johnson, president of Botany Mills, Inc, gave the commencement address on June 7th, the exercises concluding the program.

• Hamburger Delivers BDTI Commencement Address

Dr W J Hamburger, Director of Fabric Research Laboratories, Inc, Boston, Mass, delivered the Commencement Address at the Bradford Durfee Technical Institute, Fall River, Mass, on June 2, 1952. Using the theme entitled 'You are all Marked Men", Dr Hamburger outlined the responsibilities of future leaders in the textile industry in making decisions relating to encouraging, financing and utilizing scientific research. Pointing out the similarity between insurance and research, Dr Hamburger stated that, "Both insurance and research are contemplated to relieve the burdens of the future rainy day. Insurance provides cash, research provides know-how, which makes possible intelligent practical operation, preparation to meet competition, keeping ahead of competition, product development and product improvement". He went on to say that proper attitudes toward research on the part of management can only come when textile management has come to believe in resarch to the extent that "management instinctively seeks answers with an open mind and critical experimentation rather than with prejudice and opinion". Dr Hamburger cautioned BDTI graduates that they must be humble in regard to research. He said, "Science makes no policy—people make policy. Science may tell us how to do something—science cannot tell when or whether to do something. No matter what your attitude toward science may be, do not demand of science that it relieve you of the responsibility for action".

• NBTI Homecoming Weekend and Commencement

The New Bedford Textile Institute Alumni Association was again the sponsor of the annual homecoming weekend celebration of June 6, 7, and 8. Opening with registration and class reunions in the Alumni Rooms at the New Bedford Hotel on Friday, June 6, activities continued with the alumni business meeting in the morning and a Cape Cod clambake at Dan's Pavilion. An alumni golf tournament was held at Paskamansett Golf Course, with R Lake receiving the low gross award.

Among the guests of the alumni were members of the Institute's baseball team and members of the 1952 graduating class.

At the annual business meeting the Association elected the following officers:

President—Richard O Barry, Polymer Industries, Inc, Astoria, N Y; *Vice President*—Kempton Howland, Allied Chemical and Dye Corp, Providence, R I; *Treasurer*—James L Giblin, New Bedford Textile Institute; *Secretary*—Francis Tripp, New Bedford Textile Institute; *Directors for 3 years*—Edward L Murphy, Jr, Bentley Knitting Co, Providence, R I; Fred Beardsworth, New Bedford Textile Institute; and Fred R Tripp, President, Bedford Finishing Company, New Bedford, Mass.

Directors for 2 years—Howard Bates; Joseph J Crowley, U S Testing Co, Inc, Hoboken, N J; Edmund L Dupre, New Bedford Textile Institute.

Directors for 1 year—Russell Armitage, U S Testing Co, Inc, Hoboken, N J; Ralph Hawes, Apponaug Company, Apponaug, R I; James Pilkington, Riveredge Printers, Inc, Fall River, Mass.

Culminating the school year, 82 graduates of NBTI were presented degrees, diplomas and certificates at Commencement exercises in the Clarence A Cook Auditorium on June 6.

Among the award winners was Robert A Pearson, who received the award of the American Association of Textile Chemists and Colorists. Mr Pearson is to join E I du Pont de Nemours & Co, Inc.

The commencement address was given by Judge Joseph L Hurley, justice of the Massachusetts Superior Court. Following remarks to the graduates by President George Walker of NBTI, the Rev Hugh A Gallagher pronounced benediction to close the exercises.